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Development of an Identification Kit for Spilled Hazardous Materials

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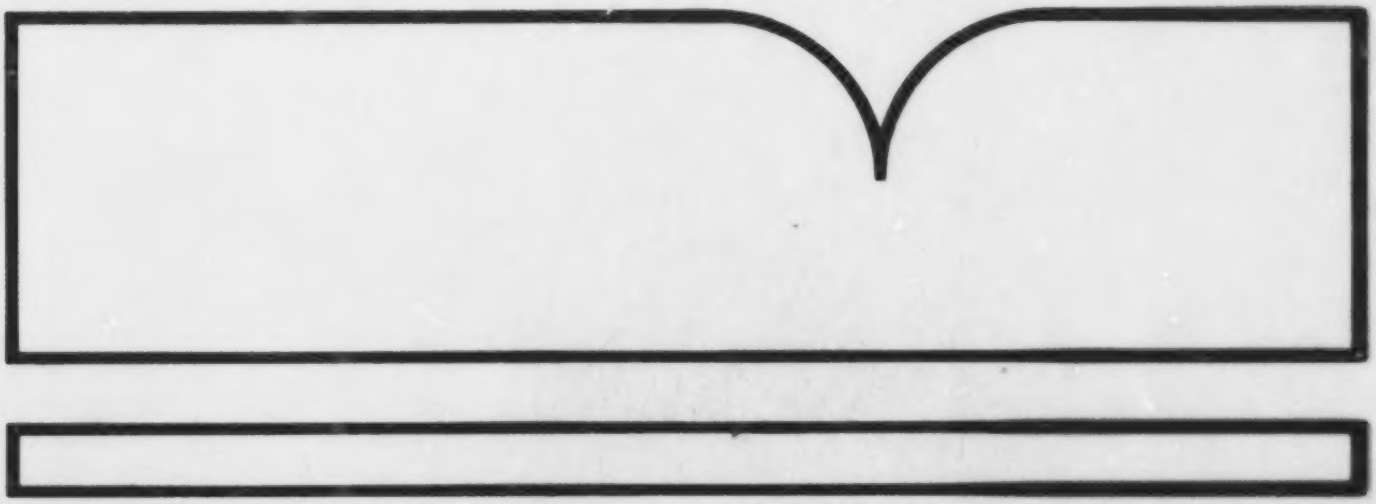

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DEVELOPMENT OF AN IDENTIFICATION KIT FOR SPILLED HAZARDOUS MATERIALS

by

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16. ABSTRACT

The Chemical Systems Laboratory (CSL) has developed a field kit to identify spilled hazardous materials in inland waters and on the ground. The Hazardous Materials Spills Identification Kit is a two-component kit consisting of an inverter/short-wave UV lamp unit for photochemical reactions and a larger package containing reagents and auxiliary equipment. The identification kit was designed as an adjunct to EPA's Hazardous Materials Detector Kit to utilize existing instrumentation, equipment, and procedures. Thirty-six materials, representative of those with the greatest hazard potential, were selected and commercial sources were screened. Procedures selected for the kit include: thin-layer chromatography, detector tubes, detector papers, CHEMets, an arsine/ Gutzeit test, and a number of color development procedures for use with the thin-layer chromatography. In addition, methods were developed for recovery of contaminants from water and soil. All information pertinent to identification of 36 specific materials was designed into a compact data retrieval system, which is included in the kit. Two prototype kits were delivered to EPA, along with a supply of consumable materials for evaluation. In addition, manuals, engineering drawings, and parts lists were provided.

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FOREWORD

The U.S. Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimonies to the deterioration of our natural environment. The complexity of that environment and the interplay of its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution; it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems to prevent, treat, and manage wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, to preserve and treat public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research and provides a most vital communications link between the researcher and the user community.

This report describes the Hazardous Materials Spills Identification Kit, which was designed as an adjunct to EPA's Hazardous Materials Detector Kit to utilize existing instrumentation, equipment, and procedures. The kit enables identification of 36 different hazardous chemicals in both water and soils by various techniques that include thin-layer chromatography, detector tubes, test papers, and CHEMets. The development of the kit is discussed, as well as the instructions for its use.

Francis T. Mayo, Director
Municipal Environmental Research
Laboratory

ABSTRACT

The Chemical Systems Laboratory (CSL) has developed a field kit to identify spilled hazardous materials in inland waters and on the ground. The Hazardous Materials Spills Identification Kit is a two-component kit consisting of an inverter/shortwave UV lamp unit for photochemical reactions and a larger package containing reagents and auxiliary equipment. The identification kit was designed as an adjunct to EPA's Hazardous Materials Detector Kit to utilize existing instrumentation, equipment, and procedures.

Thirty-six materials, representative of those with the greatest hazard potential, were selected to evaluate candidate procedures. A wide variety of methods from the literature and commercial sources were screened. Procedures selected for the kit include: thin-layer chromatography, detector tubes, detector papers, CHEMets, an arsine/Gutzeit test, and a number of color development procedures for use with the thin-layer chromatography. In addition, methods were developed for recovery of contaminants from water and soil. All information pertinent to identification of 36 specific materials was designed into a compact data retrieval system, which is included in the kit.

Two prototype kits were delivered to EPA, along with a supply of consumable materials for evaluation. In addition, manuals, engineering drawings, and parts lists were provided.

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SECTION 1

INTRODUCTION

The Oil and Hazardous Materials Spills Branch of the Environmental Protection Agency's Municipal Environmental Research Laboratory, Cincinnati (EPA) is responsible for research and development concerning control and mitigation of spills of oil and hazardous materials. Very often the nature of the spill is known; for those instances in which it is not, some method of identification is necessary in order to permit emergency response personnel to take proper action. The Chemical Systems Laboratory (CSL), under Interagency Agreement EPA-IAG-D6-0096, has developed a field kit to identify spilled hazardous materials in inland waters and on the ground. Two prototypes were fabricated and presented to EPA with drawings, parts lists, and manuals.

The program was concerned with about 500 materials listed in the Federal Register, Volume 40, Number 250, of December 30, 1975. Identification requirements of the kit and representative test materials to be used in this program were coordinated with EPA. Among others, EPA is concerned with identification of the toxic metals, anions, and classes of organic materials as follows:

Inorganics

Antimony trioxide
Arsenic trisulfide
Cadmium chloride
Lead theocyanate
Mercuric cyanide
Selenium dioxide
Zinc fluoride
Sodium arsenate
Sodium arsenite
Potassium bichromate
Potassium chromate
Potassium permanganate

Alcohols

Propyl alcohol

Aldehydes

Acetaldehyde

Nitriles

Acrylonitrile
Benzonitrile

Nitro Compounds

Nitrophenol

Organic Acids

Acetic acid
2,4-D (acid)
Maleic acid
Benzoic acid

Organic Chlorine Compounds

Dieldrin
Heptachlor
DDT
Chlorobenzene

Amines

Butylamine
Triethylamine
Aniline

Esters

Amyl acetate
2,4-D (esters)

Hydrocarbons

Isoprene
Naphthalene

Organophosphates

Diazinon
Malathion
Parathion

Phenols

Phenol

In a similar program completed in January 1976, CSL developed for EPA the Hazardous Materials Detector Kit to detect (not necessarily identify) hazardous materials in inland waters.[1] This kit is used to trace the location of contamination in water and also to warn water users until the contaminant can be treated or disappears.

EPA and CLS decided to develop an identification kit that can be used as an adjunct to the Hazardous Materials Detector Kit. This kit makes use of the pH meter, spectrophotometer, conductivity meter, and other sampling and analyzing procedures already available.

Furthermore, some of the tests used in the detector kit, if used judiciously and in combination, can provide reliable identification, particularly for inorganic materials. For example, cyanide and fluoride, which are two anions of interest, can be identified by the detector kit (Appendix, Section 4, Supplementary Identification with the Hazardous Materials Detector Kit).

Since it is planned to use the identification kit to implement corrective action in areas of high contamination, emphasis in this program was placed on reliability of identification rather than detection.

SECTION 2

CONCLUSIONS

CSL has developed a kit for use in the field to identify spilled hazardous materials in water. The design is based on the findings of an extensive investigation of literature and commercial methods in combination with laboratory studies. The operation of the identification kit has been cross-referenced and integrated with EPA's detector kit for hazardous materials.

Information pertinent to identification has been included in a compact data retrieval system, which is contained in the kit. It is possible in some cases to identify specific contaminants and, in others, to designate a class of contaminants.

The kit provides EPA with potential capability for identifying hazardous materials in the field. The kit is not simple or routine to use. However, with training and familiarization, it can provide the investigator in the field with a wide variety of information to aid him in his mission.

The kits mark a major advance over the present capability for identification of contamination in the field.

SECTION 3

RECOMMENDATIONS

The program undertaken was rather ambitious. It not only attempted to address the field identification of some 300 materials, but further sought to do so using some unconventional approaches. The research resulted in a kit that employs thin-layer chromatography, which is normally considered a rather sophisticated laboratory process, and also uses detector tubes normally used for airborne pollutants to analyze water and soil. Finally, identification is accomplished by incorporation of a data retrieval system. While every attempt has been made to anticipate all potential problems in use of this kit, it is inevitable in a program of this complexity that some difficulties will be uncovered in intensive use.

It is recommended, therefore, that EPA subject this kit to a complete series of laboratory familiarization studies to fully learn its response, handling, and overall capabilities. When this objective is accomplished, the kit should be used in a limited scope in actual field exercises. As part of the overall evaluation, EPA should consider the use concept for the kit, the extent of its application, interfaces with other detection and identification equipment, relationship to cleanup equipment and methodology, and level of skills available for use of the kit.

If it is determined that the identification kit can play a useful role, a meeting may be coordinated with CSL to review the findings. The direction, at that time, might be either of two courses. It may be expedient to identify all deficiencies at that time and implement corrections, or it may be more desirable to screen an additional group of test materials (12, 24, or 36) through the kit and then implement corrections in light of the larger data base.

In the course of making modifications or corrections, it may be desirable to investigate other nitrile tests (one was never adopted for the kit) and broader response aldehyde tests. If EPA wishes to specify a vehicle for support of this kit, it may be advantageous to set up a wiring harness under the hood leading to a common jack in the dashboard for hookup of the inverter.

Further, since all the identification objectives will not be resolved within this program, kits will be delivered to EPA at program conclusion in July 1978. These kits will provide EPA with an immediate "on-line" capability. Modifications resulting from changes in scope or shortcomings found through field experience can be made in follow-up studies.

SECTION 4

EXPERIMENTAL RESULTS

INTRODUCTION

The initial phase of this program concentrated on assembly of test methods (from literature and commercial sources) that have application to the identification of chemical contaminants in field situations. Procedures appeared numerous until they were critically evaluated by considering such factors as simplicity of operation, reliability, sensitivity, ease of maintenance, freedom from interference, etc. Unfortunately, most methods failed to meet these requirements and had to be abandoned.

Because so many methods were screened in this program, it was not possible to optimize the performance of each. It is possible that some methods that were discarded, with minor changes in pH and solvent, or other modifications, could have been used in the overall identification scheme. Nevertheless, those test methods that have been selected for inclusion in the kit have been thoroughly evaluated, are reliable, and provide a large part of the required information.

Results of testing with 36 test materials show that analytical procedures fall into the categories shown in Table 1. The intent in choosing these procedures was not only to identify the test list of materials but also to provide a range of responses that would be capable of identifying other materials from the Federal list. Identification methods are described in the following sections. A section that deals with the recovery of the contaminants from water or soil is also included.

Aqueous solutions of contaminants used in these experiments were prepared in concentrations of 1 mg per milliliter whether they were soluble to that extent or not. Tests with soil samples were made at concentrations of 1 mg contaminant per milliliter of loosely packed soil.

THIN-LAYER CHROMATOGRAPHY (TLC)

TLC procedures and methodologies were considered particularly important for identification of contaminants. In addition to providing information for class tests, in many instances, the combination of R_f value with the visualization method was able to provide specific identification. TLC studies largely concentrated on organic contaminants. Limited studies were conducted using metal reagents in combination with TLC to identify inorganic contaminants. However, it was not possible to get good resolution of the inorganics using only organic solvents for elution. The preferred aqueous systems were

TABLE 1. RESPONSE OF TEST MATERIALS TO METHODS INCLUDED IN THE IDENTIFICATION KIT

	DET PAP		CHEMets		Inorganics			Spot Tests				Detector Tubes				TLC					
	Mn^{++}	Ni^{++}	H_2O_2	Phenols	PAR	s-Diphenyl-carbazone	Dithizone	Nitro Compounds	Aldehydes	Esters	Arsine/ Gutzeit	Draeger Benzene	Draeger Olefins	BG Benzene	BG Acetone	Bromocresol G	Bromocresol G + UV	$AgNO_3$	Chloranil	$PdCl_2$	Alcohols
1. Antimony trioxide											+										
2. Arsenic trisulfide											+										
3. Cadmium chloride			±	±	+	+	+														
4. Lead thiocyanate			±	±	+	+	+														
5. Mercuric cyanide			±		±	±	+														
6. Selenium dioxide		+	±		±		+														
7. Zinc fluoride				±	+	+	+														
8. Sodium arsenate							+				+										
9. Sodium arsenite			±								+										
10. Potassium chromate	+		+		±	+	+														
11. Potassium bichromate	+		+		±	+	+														
12. Potass.permanganate	+		+	±	+	+	+														+
13. Propyl alcohol			±										+		+						
14. Acetaldehyde									+							+		+	+	±	
15. Butylamine			±																		
16. Triethylamine			±													+		+	+	±	
17. Aniline				+								+						+	+	±	
18. Amyl acetate										+					+						
19. 2,4-D (esters)										+							±	+			
20. Isoprene												+	+	+	+						
21. Naphthalene												+		+							
22. Acrylonitrile													+		+						
23. Benzonitrile																					
24. Nitrophenol				+				+													
25. Acetic acid																±		±			
26. 2,4-D (acid)												+			+	+		+	±	±	
27. Maleic acid																+		+	±	±	
28. Benzoic acid																+		+	±	±	
29. Dieldrin																	+	+			
30. Heptachlor																	+	+			
31. DDT																	+	+			
32. Chlorobenzene												+		+	+						
33. Diazinon												+		+	+			+		+	
34. Malathion														+	+	+		+		+	
35. Parathion			±											+	+	+		+		+	
36. Phenol				+														+	±		

NOTE: + = Strong Test; ± = Weak Test

considerably slower and more complex in makeup. Accordingly, it was decided to pursue more conventional methods to identify inorganic contaminants.

A variety of visualization and eluting procedures were evaluated in this program. The initial effort in the TLC study was the development of visualization techniques; once a significant number of these was selected, it was possible to go on to the development of the elution procedures.

The challenge materials were prepared in concentrations of 1 mg/ml of chloroform. Four-microliter quantities were spotted on candidate chromatographic plates; the chloroform was allowed to volatilize and then visualization was attempted. Primary emphasis was on the development of spray reagents, although other methods, such as fluorescence/quench techniques and iodine staining, were also evaluated.

Commercial spray reagents were used directly as received from the suppliers. Reagents developed in-house used Chromist sprayers (Fisher Scientific Co., Silver Spring, Maryland 20910) for application.

In addition to spray reagents developed in-house and from the literature, numerous commercially available sprays were investigated. The protocol for evaluating visualization procedures was as follows:

1. Spot 4-microliter challenge material on TLC plate.

2. Examine for long- or short-wave fluorescence and fluorescence quench.

3. Spray with candidate reagent, examine for visible color and long- or short-wave fluorescence or fluorescence quench.

4. Heat and again examine for color or fluorescence.

In addition, some reagents were irradiated for 5- to 10-minute periods with high-intensity short-wave ultraviolet light to develop colors. Other plates were exposed to iodine vapors.

Candidate procedures were evaluated on silica gel (with and without a fluorescent tracer), alumina, avicel, and cellulose chromatographic plates (Analtech, Inc., Newark, Delaware 19711). The following listing represents the major reagents that were evaluated for TLC applications. Many variations were attempted that are not listed unless the outcome was successful. Other reagents that were evaluated in similar applications but were not promising were N-ethylmaleimide[17], 1,5-dichloro-2,4-dinitrobenzene, 2-nitrophenyl hydrazine[18], p-nitrobenzene-diazoniumtetra-fluoroborate[19], potassium dichromate[14], toluene-sulfonic acid[15], 4-methylumbelliferone[10], malonic acid/salicylaldehyde, and brilliant green[15].

1. Iodine Staining[2]. Expose plates to iodine vapors by placement in a jar with a few crystals of iodine. Tan to brown spots are found with triethylamine, aniline, malathion, parathion, and phenol.

2. Fluorescence Quench. Good quenching is observed with shortwave fluorescent light with diazinon, parathion, and phenol when spotted on silica gel GF plates.

3. Indandione Reagent[3,4]. Spray with reagent; orange fluorescence is observed under shortwave fluorescent light with 2,4-D (esters), 2,4-D (acid), dieldrin, heptachlor, DDT, diazinon, and malathion on alumina plates.

4. Dragendorf Reagent (Brinkmann Instruments, Inc., Westbury, New York 11590). Spray with reagent; an orange color is observed with triethylamine on silica gel plates.

5. Iodoplatinate (Brinkmann). Spray with reagent; a purple color is observed with triethylamine on silica gel plates.

6. Rhodamine B (VWR Scientific, Inc., Baltimore, Maryland 21227). Spray with reagent; a purple color is observed with 2,4-D (acid), maleic acid, and malathion on silica gel plates.

7. 4-Dimethylaminobenzaldehyde (VWR). Spray plate and heat 2 minutes at 100°C; yellow spots are seen with aniline on silica gel and 2,4-D (acid) on cellulose and avicel plates.

8. 2',7'-Dichlorofluorescein (VWR). Spray plate and heat 2 minutes at 100°C; green spots are seen under longwave fluorescence with acetic acid, 2,4-D (acid), maleic acid, and benzoic acid on silica gel plates.

9. Molybdatophosphoric Acid (VWR). Spray plate; a blue color is seen with triethylamine on silica gel or alumina plates.

10. Bromocresol Green (VWR). Spray plate; blue spots are seen with butylamine and triethylamine and yellow spots are seen with acetic acid, 2,4-D (acid), maleic acid, and benzoic acid on silica gel plates. (See also 33 below.)

11. Mercuric Sulfate (Brinkmann). No response.

12. Ninhydrin (VWR). No response.

13. Diphenylcarbazone (Brinkmann). No response

14. Aniline Phthalate (VWR). No response.

15. Diphenylcarbazone (Gelman Instrument Co., Ann Arbor, Michigan 48106). No response.

16. Iodoplatinate (Gelman). No response.

17. Potassium Permanganate (Gelman). Spray plate; yellow spots are seen with aniline, maleic acid, and phenol on silica gel plates.

18. Ninhydrin (Gelman). No response.

19. Benzoquinone. Use as a spray at a concentration of 0.5 gm/100 ml methanol. Aniline gives a pink spot on silica gel plates.

20. Fluorescein Mercuric Acetate[5]. Use as a spray at a concentration of 17 mg/100 ml of 1N sodium hydroxide. 2,4-D (esters), heptachlor, DDT, malathion, and parathion show a quench on silica gel plates under longwave fluorescent light.

21. 4-(p-Nitrobenzyl) pyridine[2,6,7,8]. Dissolve 0.5 gm reagent, 1.0 gm mercuric cyanide, and 6.0 gm sodium perchlorate in 100 ml methanol. Spray the plate and heat 2 minutes at 100°C. Spray with a solution of 60% potassium carbonate in water. Purple spots are found with diazinon and malathion on silica gel plates and yellow spots are seen with parathion on silica gel plates.

22. 2,3-Dichloro-5,6-dicyanobenzoquinone[9]. Use as a spray at a concentration of 1.0 gm/100 ml benzene. On silica gel, triethylamine gives a red, aniline a purple, maleic acid a white, and phenol a blue color.

23. Chloranil[9]. Use as a spray at a concentration of 0.5 gm/100 ml benzene. Butylamine and triethylamine give a purple color on silica gel plates, while aniline gives a green color and phenol a peach color. All reactions require 2 minutes heat at 100°C.

24. N,2,6-Trichloro-p-benzoquinoneimine[9]. Use as a spray at a concentration of 2 gm/100 ml of methanol. Heat alumina plates for 2 minutes at 100°C. Triethylamine produces a gray spot. Acetic acid, 2,4-D (acid), and maleic acid give white spots. Malathion gives a red color and phenol a blue color.

25. 2,6-Dibromo-N-chloro-p-benzoquinoneimine[9]. Use as a spray at a concentration of 1 gm/100 ml benzene. Heat silica gel plates for 2 minutes at 100°C. Triethylamine and aniline produce black spots and phenol gives a purple color.

26. Silver Nitrate[2,6,10,11]. Use as a spray at a concentration of 0.5 gm/100 ml water. Irradiate with high-intensity ultraviolet light for 10 minutes. On silica gel plates, butylamine, triethylamine, aniline, 2,4-D (esters), acetic acid, 2,4-D (acid), maleic acid, benzoic acid, dieldrin, heptachlor, DDT, diazinon, malathion, parathion, and phenol appear as several colors, including white, gray, black, tan, and dark brown.

27. p-Phenylenediamine[12]. Use as a spray at a concentration of 0.5 gm/100 ml methanol. Irradiate 10 minutes with high-intensity shortwave ultraviolet light. On silica gel plates, triethylamine gives an orange color. 2,4-D (esters), acetic acid, 2,4-D (acid), maleic acid, benzoic acid, dieldrin, heptachlor, and malathion give purple colors; parathion gives a white color on silica gel plates.

28. Dimethylaniline[12]. Use as a spray at a concentration of 0.5 gm/100 ml methanol. Irradiate 10 minutes with high-intensity ultraviolet light. On silica gel plates, 2,4-D (esters) and 2,4-D (acid) give brown spots; dieldrin and heptachlor give purple spots, and phenol produces a white spot.

29. Diphenylamine[12]. Use as a spray at a concentration of 0.1 gm/100 ml methanol. Irradiate 10 minutes with high-intensity ultraviolet light. On silica gel plates, 2,4-D (esters) produces a purple color; 2,4-D (acid), maleic acid, and dieldrin give blue colors. Also, heptachlor gives a gray color, while parathion produces a white spot on silica gel plates.

30. Palladium Chloride[6,13]. Use as a spray at a concentration of 0.5 gm/100 ml methanol. Tributylamine, butylamine, aniline, 2,4-D (acid), maleic acid, and benzoic acid produce white to light brown spots on silica gel plates. Diazinon, malathion, and parathion give distinct brown spots on silica gel plates.

31. Tetracyanoethylene[9,14]. Use as a spray at a concentration of 0.5 gm/100 ml. Phenol appears as an orange spot on alumina plates.

32. s-Diphenylcarbazone[15]. Use as a spray at a concentration of 0.1 gm/100 ml methanol. Dieldrin, DDT, and parathion appear as red sposts on alumina plates.

33. Bromocresol Green (VWR). Spray the plate; in addition to immediate spots that develop, other materials may be detected on silica gel plates with a 10-minute irradiation with shortwave ultraviolet light. 2,4-D (esters), dieldrin, heptachlor, and DDT are found as yellow spots after irradiation.

34. Methyl Orange[16]. Use as a spray at a concentration of 0.5 gm/100 ml methanol. 2,4-D (acid) and maleic acid are seen as red spots on silica gel plates.

35. Methyl Yellow[11]. Use as a spray at a concentration of 0.5 gm/100 ml methanol. Butylamine, triethylamine, aniline, diazinon, malathion, and parathion give yellow colors on silica gel plates, while acetic acid, 2,4-D (acid), and maleic acid give red spots. also, if the plate is irradiated with shortwave ultraviolet light, blue spots are seen with acetic acid, 2,4-D (acid), and benzoic acid.

36. o-Toluidine[12]. Use as a spray at a concentration of 0.5 gm/100 ml methanol. Irradiate 10 minutes with shortwave ultraviolet light. Aniline, 2,4-D (esters), 2,4-D (acid), and maleic acid appear as brown spots on silica get plates, while parathion appears white.

37. 2,2-Diphenyl-1-picrylhydrazyl[15]. Use as a spray at a concentration of 0.1 gm/100 ml chloroform. On silica gel plates at 100°C for 2 minutes, butylamine, 2,4-D (acid), maleic acid, malathion, and phenol produce yellow spots, while aniline gives a blue color.

38. N,N-Dimethyl-p-phenylenediamine dihydrochloride[15]. Use as a spray at a concentration of 0.5 gm/100 ml methanol. On silica gel plates with shortwave ultraviolet radiation, butylamine, triethylamine, and aniline appear as gray spots.

Materials visualized are from the selected list of contaminants. A summary of findings of the most significant visualization procedures is given in Table 1 and also in Section 5, Identification Concept. Detailed use of these methods is described in the Appendix, Section 3, Analytical Procedures.

From the initial screening it was concluded that the following reagents were the most promising for exploitation of TLC procedures:

Iodine vapors
Indandione
Bromocresol green with and without UV light irradiation
Methyl yellow with and without UV light irradiation
2',7'-Dichlorofluorescein
4-(p-Nitrobenzyl) pyridine
Palladium chloride
Benzoquinone
Chloranil
2,3-Dichloro-5,6-dicyanobenzoquinone
2,2-Diphenyl-1-lpicrylhydrazyl
Silver nitrate
Diphenylamine
Dimethylaniline
o-Toluidine

Continued testing showed some of the reagents to be unstable, unreliable, complex, or insensitive. Furthermore, some, which were based on fluorescence readout, were subject to interference by the recovery methods developed for contaminants (see Recovery and Cleanup, this section).

Accordingly, a final selection was made for the kit that included bromocresol green (with and without UV irradiation), silver nitrate, palladium chloride, and chloranil. Findings with these reagents are presented in Table 1. These are considered to be the easiest to use and most reliable and stable reagents. Furthermore, they detect a good cross-section of materials and nalyxhibit a degree of overlap that enables them to act as backup in some analyses. Using these procedures, it is possible to detect 16 of the 24 organic contaminants used in this program. The procedures detect amines, acids, chlorinated materials, thiophosphates, and phenols.

In the course of this investigation, numerous reagents were developed that are ideally used in a spray form. The method is attractive for visualizing unknowns by the TLC procedures described and also for detecting certain inorganic contaminants (see Inorganics Spray Reagents, this section). Accordingly, a contract was let with Case-Mason Filling, Inc., of Joppa, Maryland 21085, to package the reagents into self-contained pressurized cans. The reagents, formulation, and use are given in Table 2.

TABLE 2. CANDIDATE REAGENTS FOR PRESSURIZED SPRAY CANS

Reagents	Use	Formulation
Silver nitrate	TLC - Organics	5.0 gm/l water
Palladium	TLC - Organics	5.0 gm/l methanol
Chloranil	TLC - Organics	5.0 gm/l chloroform
Diphenylamine	TLC - Organics	1.0 gm/l methanol
Alizarin	Inorganics	0.5 gm/l methanol
4-(2-Pyridylazo) resorcinol	Inorganics	0.65 gm/l methanol
Xylenol orange	Inorganics	2.5 gm/l methanol
s-Diphenylcarbazone	Inorganics	5.0 gm/l chloroform
Diphenythiocarbazone (Dithizone)	Inorganics	0.5 gm/l chloroform

The packager prepared all reagent/propellant mixtures in clear glass containers, which made it possible to determine interactions when they occurred. The propellant of choice was an equal mixture of Freon-11 and Freon-12. Sometimes, the propellant precipitated the reagent out of solution. When this happened, a second propellant, "A46," a mixture of isobutane and propane designed to produce pressure of 46 psi, was used.

Alizarin red-S and xylenol orange precipitated regardless of which propellant was used; silver nitrate attacked the metal can and could not be packaged. All the other reagents were prepared in the Freon mixture and returned to the laboratory for evaluation. Shortly thereafter, all the reagents, with the exception of PAR and chloranil, decomposed to the point where they could not be used. Chloranil, probably due to its low solubility, tended to plug the tip of the spray can, eventually rendering it useless. The self-contained PAR spray can was selected for use in the kit. Its composition is as follows:

Can: 6 oz Spraytainer, plain

Button: Seaquist 3125 Narrow Spray, forwardlook style, mechanical breakout

Valve: Sea-Spray, NS-34, 0.018-inch stem, dimpled mounting cap, Buna-N gasket, 0.023-inch wire stainless steel spring, capillary body orifice, tubed 4-1/16-inch, 0.060-inch capillary I.D.

Fill: 80 gm Freon 11
80 gm Freon 12
40 gm PAR 0.63 gm/l methanol

Other spray reagents that are of interest to the kit are packaged independently of the propellant system. The reagents are provided in a glass jar

and a Chromist sprayer is attached as needed.

Once adequate visualization procedures were available, it was possible to develop elution procedures for TLC. Because TLC would be applicable to those contaminants that were capable of visualization, all subsequent elution studies were limited to these 16 materials (see Figures 1 through 4 and Table 3). Also, because certain variables, such as plate quality, temperature, humidity, solvent purity, etc., were expected to affect the elutions, it was decided to include some standard reference dyes in the separations. Out of a number of dyes considered, indophenol, sudan orange R, and sudan yellow 3G were determined to produce the most compact, uniform, and reproducible movements. Sudan yellow 3G was ultimately chosen for the kit; even with use of a reference dye, variations are found in the elution data. However, the dye serves as a good indicator of overall performance of the system in the field.

Separations were made using prescored 10-cm alumina and silica gel plates (Analtech, Inc.); this size was determined to be the minimum that would produce acceptable resolution. Screening of elution solvents was made using an eulotropic series that included acetonitrile, methanol, acetone, isopropanol, ethyl acetate, chloroform, dichloromethane, benzene, toluene, and hexane.

It was concluded from this initial screening that silica gel plates were sturdier than alumina for field use and resolutions were superior. Also, chloroform produced varied separations which would be more useful in applying the process to identification. Acetone, while not as good a separator as chloroform, could also be useful.

In the course of this evaluation, Analtech, Inc., started marketing of a hardened silica gel plate. This plate was extremely rugged and could be written on, thus it was decided to evaluate it more extensively. Elution data were derived by the following procedure:

1. A rectangular tank for 20- x 20-cm plates was used for elution. The sides and back were lined with glass fiber paper in order to quickly and more evenly saturate the atmosphere.

2. Enough solvent was added to the tank to wet the glass fiber and give a depth of 3 to 5 mm.

3. Four microliters of the materials to be chromatographed (1 mg/ml in chloroform) were spotted 10 mm from the lower edge of the chromatographic plate. The material was spc ed in several applications in order to maintain a compact spot.

4. Marks were made on the upper side edges of the plate 70 mm from the spots (80 mm from the bottom of the plate).

5. The plate was made to stand upright and the top was placed on the tank.

6. When the solvent front reached the upper markings, the plate was removed and allowed to dry.

7. Visualization was accomplished by the techniques described earlier and the locations of contaminants were marked.

8. R_f values--distance traveled by contaminant divided by distance traveled by eluting solvent (70 mm) -- were calculated.

9. On some occasions, R_s values were calculated. These were distance traveled by contaminants divided by distance traveled by standard dye.

Separations made on silica gel Hard Layer (Organic Binder) TLC Uniplates are depicted in Figure 1 in a standard laboratory tank using procedures described above; separation and R_f and R_s data are given in Table 3.

In order to place TLC in a format that would be acceptable for field use, smaller chromatographic tanks to replace the larger laboratory type were sought. A small tank (source unknown) measuring 21-1/2 cm by 11 cm by 5 cm was ideally suited for the 10-cm by 20-cm chromatographic plates. When glass fiber paper lining was used in the tank, results were reproducible and closely agreed with data derived with the larger tank. Unfortunately, an extensive search for the source of this tank was unsuccessful and had to be abandoned.

As an alternate choice, a number of commercial sandwich chambers were investigated. They, again, were compact, lightweight, and were claimed to generate reproducible data. Three chambers that were investigated were found to be poorly designed, difficult to assemble, or not suited for use of glass plates.

A successful chamber design was prepared in-house. (Appendix, Section 2, Equipment and Methodology.) Problems were first experienced with this chamber when the separator used between the two plates was 1905 microliters. When this thickness was reduced to 762 microliters, results became more reproducible.

Figure 2 depicts separations at room temperature made with the sandwich chamber proposed for the identification kit. The separation and R_f and R_s values are presented in Table 3. Notice that some changes observed over the standard laboratory tank are quite dramatic.

It was long suspected in these investigations that certain factors particularly affected the movement of materials on chromatographic plates. Temperature was considered to be one of the most important factors. Accordingly, the separations made in the sandwich chamber were rerun in an environmental chamber. In Figures 3 and 4 the effects of temperature on the separations are seen. Again, the separation and R_f and R_s values are presented in Table 3.

It is evident from the above analysis, Figures 2 through 4, and Table 3, that even the use of standard dyes is not sufficient to produce consistent R_f and R_s values under all conditions. All materials are not similarly affected by temperature. The elution rates of contaminants may be more sensitive to temperature than the standard dyes. Therefore, unless separations are made at similar temperatures, the use of standard dyes primarily serves to indicate the general overall operability of the system in the field.

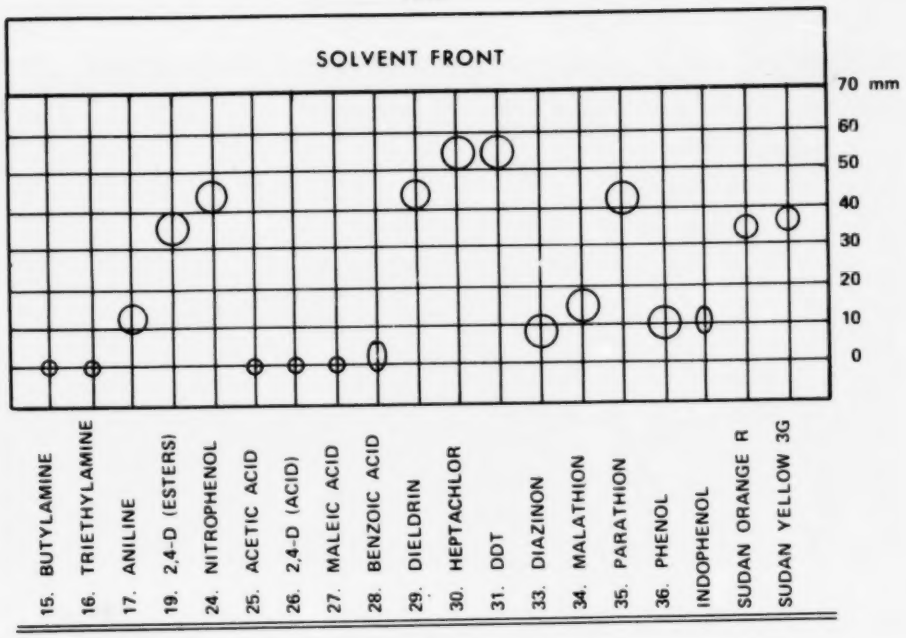

Figure 1. Elution of organic contaminants and standard dyes with chloroform on silica gel HL plates in a standard tank at room temperature.

In summation, it is concluded that TLC can serve a highly useful purpose in the identification kit. Accordingly, a sandwich chamber, using hard-layer silica gel plates and chloroform solvent, was selected. The qualitative use of R_f and R_s values in combination with recommended visualization techniques provided a substantial amount of information for identification of organic contaminants.

DETECTOR TUBES

The procedure for applying detector tubes for airborne contaminants to water testing has been described.[1] The process is also presented in detail in the Appendix, Section 2, Equipment and Methodology. It consisted of placing water suspected of contamination into a small jar. An effervescent material, e.g., bicarbonate, was added and the jar was capped with a detector tube affixed to the cap so that the emitted gases were forced through the tube. The tube was noted for a color response due to the evolved contaminant.

The method described above can be modified for soil testing by the following procedure. An equivalent of 40 ml of loosely packed soil can be added to the effervescent jar. Add distilled water to the 80-ml mark and stir the mixture for several minutes. Add effervescent material to the mix, cap the jar, and complete the test as described above.

TABLE 3. R_f AND R_s VALUES OF ORGANIC CONTAMINANTS AND STANDARD DYES ELUTED WITH CHLOROFORM ON SILICA GEL HL UNIPLATES

	Standard Tank			Sandwich Chamber								
	Room			30-40°F			Room			90-100°F		
Test Material	mm Traveled	R_f	R_s	mm Traveled	R_f	R_s	mm Traveled	R_f	R_s	mm Traveled	R_f	R_s
15. Butylamine	0	0.00	0.00	0	0	0	0	0	0	0	0	0
16. Triethylamine	0	0.00	0.00	0	0	0	0	0	0	0	0	0
17. Aniline	12	0.18	0.36	10	0.14	0.37	23	0.33	0.46	14	0.20	0.41
19. 2,4-D (Esters)	37	0.53	1.08	32	0.46	1.19	49	0.70	0.98	40	0.57	1.18
24. Nitrophenol	43	0.61	1.24	49	0.70	1.81	58	0.83	1.16	51	0.73	1.50
25. Acetic Acid	2	0.03	0.06	3	0.04	0.11	5	0.07	0.10	4	0.06	0.12
26. 2,4-D Acid	2	0.03	0.06	7	0.10	0.26	6	0.09	0.12	5	0.07	0.15
27. Maleic Acid	0	0.00	0.00	1	0.01	0.04	0	0.00	0.00	1	0.01	0.03
28. Benzoic Acid	5	0.08	0.16	8	0.11	0.30	9	0.13	0.18	8	0.11	0.24
29. Dieldrin	44	0.62	1.28	52	0.74	1.93	63	0.90	1.26	55	0.79	1.62
30. Heptachlor	54	0.78	1.60	70	1.00	2.59	70	1.00	1.40	70	1.00	2.06
31. DDT	54	0.78	1.60	70	1.00	2.59	70	1.00	1.40	70	1.00	2.06
33. Diazinon	8	0.12	0.24	8	0.11	0.30	19	0.27	0.38	10	0.14	0.29
34. Malathion	16	0.22	0.45	8	0.11	0.30	23	0.33	0.46	12	0.17	0.35
35. Parathion	41	0.58	1.21	41	0.59	1.52	53	0.76	1.06	43	0.61	1.26
36. Phenol	9	0.13	0.27	8	0.11	0.30	11	0.16	0.22	10	0.14	0.29
Indophenol	10	0.14	0.27	8	0.11	0.30	21	0.30	0.42	9	0.13	0.26
Sudan Orange R	33	0.47	0.94	29	0.41	1.07	50	0.71	1.00	35	0.50	1.03
Sudan Yellow 3G	35	0.50	1.00	27	0.39	1.00	50	0.71	1.00	34	0.49	1.00

Note: Solvent travel is 70 mm.
R_s = Normalized to sudan yellow 3G.

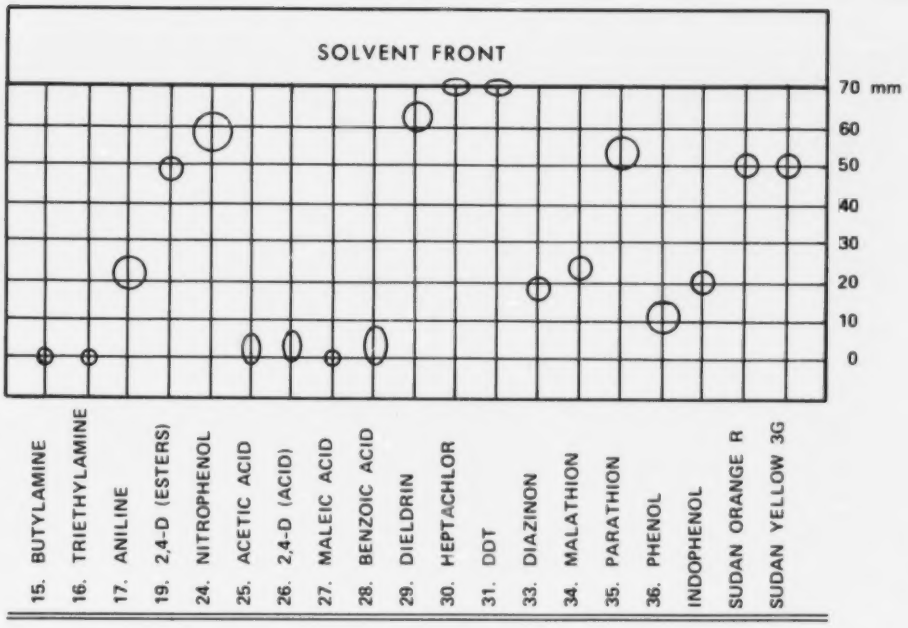

Figure 2. Elution of organic contaminants and standard dyes with chloroform on silica gel HL plates in a sandwich chamber at room temperature.

Soils having a high organic content will often cause excessive foaming, which carries the water into the detector tube. It is necessary in these cases to provide a small ballast chamber between the cap of the jar and the detector tube to allow the foaming to subside.

A survey was made of the tubes of the major suppliers (Kitagawa: Matheson Gas Products, Lyndhurst, New Jersey 07071; Bendix/Gastec: National Environmental Instruments, Inc., Warwick, Rhode Island 02888; Draeger: National Mine Service Company, Pittsburgh, Pennsylvania 15219; and Mine Safety Appliances, Pittsburgh, Pennsylvania 15235). Available data on detection reagents, detected materials, probable interferences, and sensitivity were catalogued. It was seen from the survey that many detection systems were repetitive, not only among companies, but also within the same company. For example, many companies carry various tubes containing chromium salts and sulfuric acid that are designated for detection of certain compounds. One company alone carries 24 variations of this tube, which is used to detect 24 different organic materials. It is believed that some tubes are not as selective as may be implied.

From the survey, 24 types of tubes were selected for evaluation as being highly representative of those available. In the initial screening, the tubes were evaluated against aqueous solutions containing 1 mg/ml of the challenge contaminants. Most tubes were discarded because they were not sufficiently sensitive or else produced too many borderline responses that were difficult to classify. Promising tubes were further evaluated against natural waters and soils.

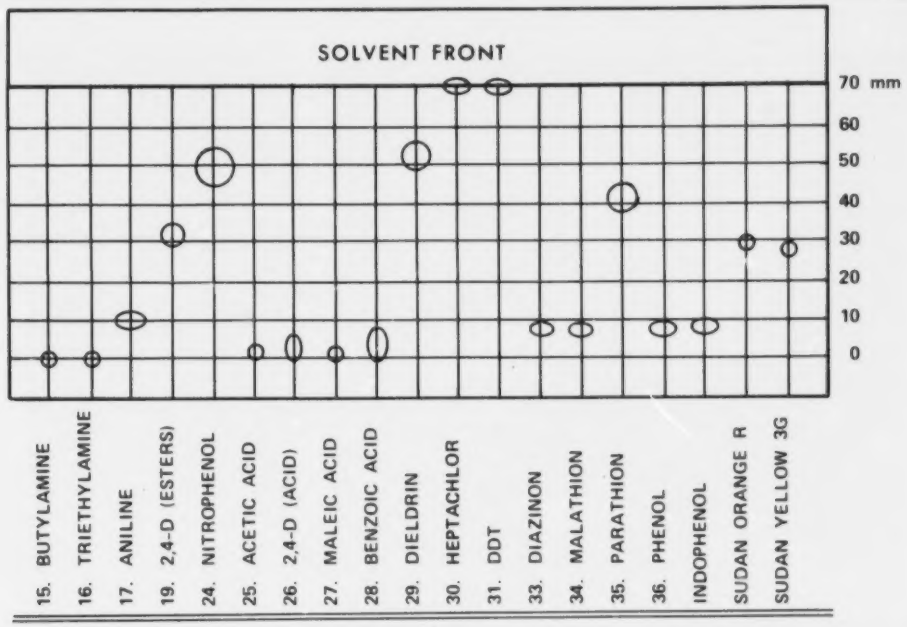

Figure 3. Elution of organic contaminants and standard dyes with chloroform on silica gel HL plates in a sandwich chamber at 30-40°F

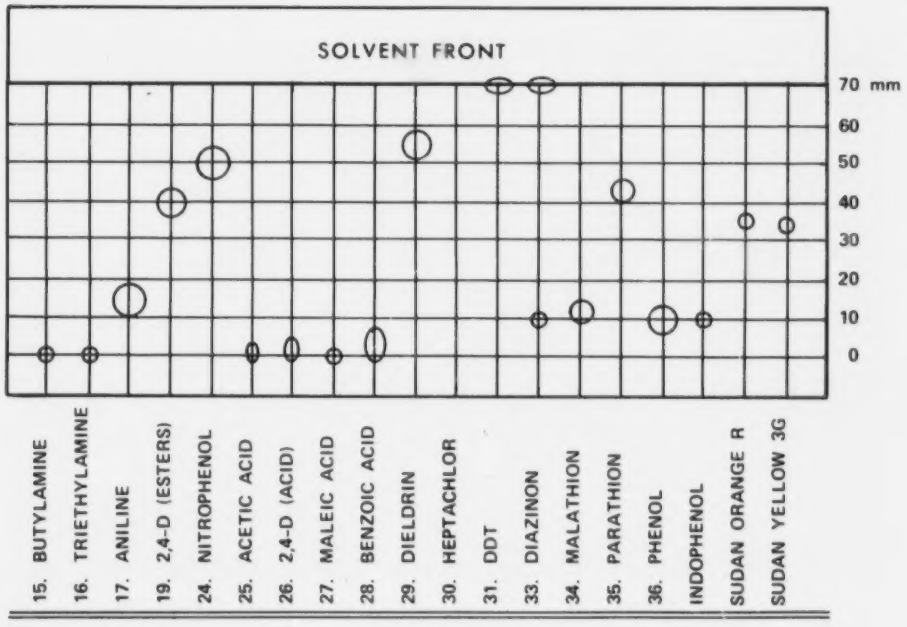

Figure 4. Elution of organic contaminants and standard dyes with chloroform on silica gel HP plates in a sandwich chamber at 90-100°F

The tubes selected for inclusion in the kit were Benzene 5/a, Olefin 0.05%/a, Benzene No. 121, and Acetone No. 151. Findings are summarized in Table 1. The initial screening details are summarized as follows:

1. Arsine 0.05a (Draeger). A light blue color forms on the precleanse layer with malathion.

2. Formaldehyde 0.002 (Draeger). A brown color forms on the indicating layer with isoprene. Acetaldehyde gives a reddish color on the indicating layer.

3. Aniline 5/a (Draeger). A pink color is obtained on the indicating layer with aniline.

4. Benzene 5/a (Draeger). Purple colors form on the precleanse and indicating layers with aniline. Isoprene turns the precleanse layer brown; naphthalene colors the precleanse layer blue; 2,4-D (acid) turns the precleanse layer a reddish-brown; and chlorobenzene turns the precleanse layer pink. Malathion turns the precleanse layer light brown and the indicating layer pink. Diazinon produces a reddish-violet color on the precleanse layer.

5. Carbon disulfide 30/a (Draeger). Nitrophenol produces a yellow color on the indicating layer.

6. Carbon tetrachloride 5/c (Draeger). Isoprene turns the indicating layer green and causes the reagent in the ampoule to turn brown. Chlorobenzene produces a light brown color with ampoule reagent.

7. Olefin 0.05%/a (Draeger). Light brown colors are observed on the indicating layer with acetaldehyde, isoprene, and acrylonitrile.

8. Polytest (Draeger). A brown color forms on the indicating layer with aniline, amyl acetate, and isoprene. Naphthalene produces a blue color and chlorobenzene produces a brownish-purple color. Acetaldehyde and butylamine give brownish-green colors on the indicating layer.

9. Perchloroethylene 10/a (Draeger). A blue color is produced on the indicating layer with chlorobenzene, malathion, parathion, and phenol.

10. Trichloroethane 50/b (Draeger). Gives positive tests with water; was abandoned.

11. Hydrogen fluoride 1.5b (Draeger). Negative response.

12. Cyanogen chloride 0.25/a (Draeger). Negative response.

13. Systox 1/2 (Draeger). Negative response.

14. Ammonia 25/a (Draeger). Negative response.

15. Toluene diisocyanate 0.2/A (Draeger). Negative response.

16. Trichlorethylene 10/a (Draeger). A brown color is obtained with isoprene and acrylonitrile on the precleanse layer. Chlorobenzene gives an orange color on the indicating layer.

17. Phenol 5/a (Draeger). A pink color is obtained with butylamine on the indicating layer.

18. Styrene No. 124 (Bendix/Gastic). A yellow color forms on the indicating layer with isoprene.

19. Ethylene No. 172 (Bendix/Gastec). Isoprene and malathion produce a dark blue color on the indicating layer.

20. Carbon tetrachloride No. 134 (Bendix/Gastec). Isoprene and chlorobenzene give dark brown colors on the indicating layer.

21. Carbon monoxide 1LL (Bendix/Gastec). Malathion produces a brown color on the indicating layer.

22. Benzene No. 121 (Bendix/Gastec). Mixtures of colors, including yellows, oranges, greens, browns, and grays, are obtained with isoprene, naphthalene, heptachlor, DDT, chlorobenzene, diazinon, malathion, parathion, and phenol.

23. Acrylonitrile, No. 191 (Bendix/Gastec). Isoprene gives a brown color on the indicating layer; naphthalene gives a pink color on the indicating layer. Chlorobenzene produces a black on the precleanse layer and a pink on the indicating layer. Diazinon gives a yellow-green on the precleanse layer and a pink on the indicating layer.

24. Acetone No. 151 (Bendix/Gastec). A mixture of colors, including green, brown, and black, is obtained with amyl acetate, isoprene, acrylonitrile, acetic acid, chlorobenzene, and diazinon, on the indicating layer.

ARSINE/GUTZEIT

One of the most reliable methods for detection and identification of arsenic is the classic Gutzeit test[20]. In this procedure, zinc metal and strong acid are added to an aqueous solution of an arsenical compound (the procedure is effective for inorganic and certain aliphatic arsenicals). Nascent hydrogen, which is formed, reacts with he arsenic to form arsine. Arsine can be detected by test papers impregnated with various mercuric compounds.

In the procedure developed for the kit, Draeger Tube AsH_3 0.05/a was substituted for the test paper. The assembly described above for the detector tube tests was used. In the initial evaluation of the system, hydrogen gas was generated by adding 1 gm (scoop measure) 30-mesh zinc metal and 1 gm

(scoop measure) potassium bisulfate with a crystal of cupric sulfate for catalyst to 80 ml of test solution. Evolved arsine gas turned the detector tube a grayish-purple. The procedure was effective for all the arsenical compounds on the challenge list of materials. It was even useful for antimony compounds. Some difficulties were experienced with antimony trioxide due to its low solubility, but when more soluble antimony compounds were tested, strong responses were obtained.

Midwest Research Institute, Kansas City, Missouri 64110, under contract to the Chemical Systems Laboratory for another program, studied the use of a Gutzeit mix (zinc, potassium bisulfate, and cupric sulfate) in detection of certain chemical agents. In their studies, they prepared a formulation packaged in compact trilaminate plastic pouches; this preparation was more sensitive and easier to handle than the individually measured quantities of chemicals used in this program. Accordingly, a supply of Gutzeit pouches was purchased and evaluated.

Use of the Midwest Gutzeit preparation showed a marked increase in sensitivity, wherein all the antimony and arsenical materials on the challenge list were detected. Midwest's preparation has been included in the identification kit.

The above procedure may be used for soil testing by adding about 40 ml of loosely packed soil to the effervescent jar and diluting to the 80-ml mark with water. The procedure for water testing is then followed. Details may be found in the Appendix, Analytical Procedures. Findings are presented in Table 1.

SPOT TESTS

Identification of some classes of materials was not achieved with TLC or detector tubes, namely alochols, aldehydes, esters, hydrocarbons, nitriles, and nitro compounds. This area is particularly difficult as there are no commercial tests available for these materials.

An extensive screening of spot test procedures for identification of these compounds was carried out. The areas investigated are described in the following sections.

Nitro-Containing Compounds

A very promising test for nitro-containing compounds, which uses a 2-amino alcohol as a colorimetric reagent[21], was developed. Reagents tested were ethanolamine, 2-amino-2-methyl-1-propanol, 2-amino-2-methyl-1, 3-propanediol, and 2-(2-aminoethyl-amino)-ethanol. These reagents, used neat, all give a bright yellow color with an aqueous solution (1 mg/ml) of nitrophenol. Furthermore, these reagents appear to be highly specific for the nitro group of a large number of aromatic nitro compounds as well as for some aliphatic nitro compounds, giving yellow, orange, red, brown, and blue colorations. The reagents and nitro-containing materials were used neat except when both compounds were solids, in which case several drops of methanol were added.

The colors obtained with ethanolamine (1-amino-2-ethanol) and other nitro-

containing compounds from the Federal Register are shown below.

1,3-Dinitrobenzene ------- deep yellow
2,4-Dinitrophenol ------- red
Nitrobenzene ------------ yellow (on standing)
Nitrophenol ------------- deep yellow
Parathion --------------- no color

None of the other organic compounds in the list of challenge materials gave a positive test with ethanolamine.

Yagoda Confined Spot Test Papers, carried by Schleicher and Schuell, Inc., Keene, New Hampshire 03431, were found to be an effective and inexpensive method for conducting spot tests. The papers, which measure 40 by 40 mm, have 1-cm test spots contained by a ring of inert impervious waxlike material. The test material is applied to the center spot followed by the reagent. The procedure was highly effective in detecting nitrophenol with ethanolamine.

One-percent methanolic solutions of ethanolamine were also studied, but the concentrated form was more sensitive and, therefore, preferred.

The procedure was modified for the kit as follows: To one drop of the water solution on a segment of silica gel HL Uniplate, add one drop of ethanolamine. A deep yellow color is produced by nitro compounds. Compare with a distilled water blank, if necessary.

The test for nitro compounds using ethanolamine was also found to be effective with water extracts of soil samples. The findings are presented in Table 1.

Aldehydes

A deep red color developed when one drop of 5% sodium nitroprusside (Nitroferricyanide, $Na_2Fe(CN)_5NO \cdot 2H_2O$) and one drop of a saturated solution of sodium hydroxide in methanol were added to 1 ml of aqueous test solution (1 mg acetaldehyde/ml water)[22]. None of the other organic materials responded with sodium nitroprusside.

When the test was carried out using a chloroform solution of acetaldehyde, the aqueous nitroprusside solution separated to the top. However, on shaking, a red color concentrated in the aqueous layer.

Bases other than sodium hydroxide were investigated; organic bases are reported to give a blue color with acetaldehyde. If one drop of piperidine is substituted for sodium hydroxide, a deep blue-violet color is obtained.

Neither formaldehyde nor paraformaldehyde, which are on the comprehensive list of organic contaminants, responded to the sodium nitroprusside test. It may be too narrow as a class test for aldehydes; however, it was included in the kit because of its excellent sensitivity. The test was modified for the kit as follows: To 1 ml of the water solution, one drop of 5% sodium nitroprusside in water and one drop of saturated sodium hydroxide in methanol were added. A deep red color was observed with acetaldehyde. The procedure was

also effective with water extracts of soil samples. Findings are presented in Table 1.

Esters

The determination of esters is based on reaction of an ester with hydroxylamine in alkaline solution to form hydroxamic acid.[23] Hydroxamic acids give a violet-colored chelate with ferric ion. Some difficulties were encountered in repeating the tests. There were apparent stability problems and the proportions of reagents needed to be investigated.

The procedure first developed produced a white precipitate with the addition of hydroxylamine and sodium hydroxide to the test solution, which tended to obscure color formation. The precipitate was sodium chloride, which is insoluble in the solvents used (methanol and chloroform). This problem was eliminated by running the test in a water solution; the sodium chloride dissolves and does not interfere.

Another difficulty was found in the reproducibility of the test; it appears that the disposable plastic tubes used for testing were a source of interference. When the work was repeated using glass vials, the results were consistent.

It was further found that when the reaction mixture is heated in a boiling water bath, deep purple colors are obtained. However, prolonged heating causes decomposition of some hydroxamic acids and, therefore, should be avoided. After heating, 5% hydrochloric acid is used to neutralize the excess sodium hydroxide before adding the ferric ion. The amount of acid added is critical. If not enough is added, ferric hydroxide will precipitate. On the other hand, too much acid will hinder color development. The optimum pH is about 3 to 6. In order to provide for pH adjustment, sodium alizarin sulfonate indicator was added to the hydrochloric acid.

Hydroxylamine hydrochloride and sodium hydroxide reagents were prepared in methanol and compared with similar reagents in water; the methanolic reagents were found to be more sensitive and are recommended. The hydrochloric acid/sodium alizarin sulfonate reagent was modified by increasing the acid content from 5% to 10%, which reduces the volume of reagent required for the test. Heat is no longer found necessary for the test. The test, as modified, detects both amyl acetate (deep purple color), and 2,4-D (esters) (brownish-amber color), although it is more sensitive to the former.

The procedure is also effective for detecting esters in water extracts of soils. The procedure in the kit is as follows:

> To 1 ml of test water in a test tube, add 0.5 ml (10 drops) of 12.5% hydroxylamine hydrochloride in methanol and 0.5 ml (10 drops) of 12.5% sodium hydroxide in methanol. Allow to stand 2 to 3 minutes. Acidify with 10% hydrochloric acid solution containing 0.01% sodium alizarin sulfonate (the solution turns purple to red to orange to yellow). Stop acid addition when the solution just turns yellow. Add two drops of ferric chloride solution (5 gm $FeCl_3$ and five drops HCl in 50 ml water). Esters

may give a color ranging from brownish-amber to purple. Compare with a water blank if there is any doubt.

Findings are presented in Table 1.

Alcohols

Vanadium(V)-8-hydroxyquinoline complex[24] was studied as a reagent for alcohols. A red color was obtained with alcohols in organic solvents. (The test was not effective in aqueous solutions.) It was first proposed to run the test in chloroform since this solvent, in conjunction with XAD-2 amberlite cartridges, was selected for recovery of contaminants from water (see Recovery and Cleanup). However, chloroform was found to contain traces of alcohol and therefore, all test solutions produced positive blanks. Consideration was given to running the test in methylene chloride since this was an alternate solvent to chloroform for recovery. However, methylene chloride was found to extract interfering materials from the XAD-2 column and could not be used for water testing. Prior washing of the column with methylene chloride failed to remove the interferences.

An alternate method was to extract the alcohol from water directly by solvent. The test solution was shaken with a toluene solution of vanadium oxinate and acetic acid. When the test tube was heated in a water bath, a faint brownish-pink color was obtained; sensitivity was inadequate for kit purposes.

Surprisingly, when the vanadium test was applied to soil testing, alcohol was easily detected in the methylene chloride, which was used for extraction. This was true even in soils having a high organic content. Accordingly, the test was adopted for kit use, but limited to soil analysis. the procedure used was as follows:

> To 1 ml methylene chloride extract of the contaminant, add two drops of 1% vanadium(V)-8-hydroxyquinoline complex and two drops of glacial acetic acid. After 1 minute, a deep red color develops in the presence of alcohols.

Findings are presented in Table 1.

Use of diammonium ceric nitrate as a reagent for alcohol was also investigated. This reaction would be ideal since it can be carried out in water; unfortunately, the sensitivity is not adequate. Various organic acids and salts (HCl, $ZnCl_2$, $SnCl_4$, and AlF_3) were added to the reaction medium to try to increase the sensitivity, but nothing showed promise.

Nitriles

Tests reported in the literature for detection of the -CN group are based on pyrolysis at temperatures of 180°C to 250°C (a technique not practical for field testing), or they use a method that is not specific for nitriles.

Experimental work showed that acrylonitrile and benzonitrile form colored compounds with certain aromatic nitrocompounds (e.g., m-dinitrobenzene,

nitrofluorene). These compounds did not give consistent results.

Nitriles react with hydroxylamine to produce amidoximes, which usually give red colors with ferric ion. Preliminary investigation of this reaction showed that acetonitrole gives a brown-red color and benzonitrile gives a green color. Generally, higher heating conditions are required; a method of adapting the test to kit use was not found.

CHEMETS

CHEMetrics Kits (CHEMetrics, Inc., Warrenton, Virginia 22186) were screened as tests for hazardous materials. CHEMets are disposable glass ampoules, about the size of a cigarette, used for the colorimetric analysis of water samples. Each CHEMet contains a color-forming reagent sealed under vacuum. When the tip of the CHEMet is broken under water, water rises into the ampoule and dissolves the reagent. A color forms if the sought compound or ion is present. A color comparator composed of liquid standards is offered for quantitative measurement.

CHEMets were screened against aqueous solutions containing 1 mg/ml of the challenge contaminants. Most CHEMets were found not be be sufficiently responsive to the test materials. Two, however, that passed this initial and subsequent screenings with natural waters and soils are phenols - Model P-12, and hydrogen peroxide - Model HP-10; they are included in the identification kit.

Findings are presented in Table 1 and CHEMets screened in this program are shown below.

1. Phenols, Model P-12. Gives excellent response to phenol. Nitrophenol and aniline are also detected. Light precipitates are obtained with cadmium chloride, lead thiocyanate, zinc fluoride, and potassium permanganate.

2. Hydrogen peroxide, Model HP-10. Gives dark brown colors with potassium bichromate, potassium chromate, and potassium permanganate. Weaker brown or yellow colors and light precipitates are obtained with cadmium chloride, lead thiocyanate, mercuric cyanide, selenium dioxide, sodium arsenite, propyl alcohol, butylamine, triethylamine, and parathion.

3. Phosphate, Model PO-150. Excellent response to inorganic phosphate, but does not detect phosphate esters, diazinon, malathion, or parathion.

4. Hydrazine, Model H-5. Detects aniline.

5. Iron, Model Fe-10. Detects only iron.

6. Dissolved oxygen, Model O-12. Detects oxygen.

7. Ammonia nitrogen, Model AN. Response to acetaldehyde and potassium permanganate.

8. Chlorine, Model Cl-2. Does not detect any of the organic chlorine compounds. Sensitive response to potassium permanganate.

DETECTOR PAPERS

Most of the reagents considered for inorganic contaminants were initially evaluated by impregnating them on test paper dip strips. Literature procedures were screened with the view of finding chemicals that could be adapted to a paper similar to a pH paper, which would give a specific color response to a particular metal or ion. The concept is appealing because it offers a high volume of tests in a compact, inexpensive, and easy-to-use configuration.

A number of papers were impregnated with chemical chromogenic reagents. The general method used was to wet filter paper (9-cm circles of Whatman #40) with a 1% methanolic solution of the reagent, air-dry the paper, and then cut it into strips. Testing was accomplished with aqueous solutions of contaminants at concentrations of 1 mg/ml, whether or not they were soluble to that extent.

E.M. Quant Strips (Scientific Products, Division of American Hospital Supply Corp., McGraw Park, Illinois 60085), test papers for water testing, were also evaluated. (Directions for use of the strips specify adjustment of pH or other modification of water samples to make the tests specific. However, they were evaluated with untreated waters as were the in-house prepared strips.)

The following detector paper systems were screened in this program:

Al^{+3} (Quant strip)	Rhodamine B
Fe^{+3} (Quant strip)	8-Hydroxyquinoline
$Chromate^{+3}$ (Quant strip)	Thionalide
Ether peroxide (Quant strip)	as-Diphenylhydrazine
Zn^{+2} (Quant strip)	PAR (4-(2-Pyridylazo)-resorcinol)[25]
Sulfite (Quant strip)	1,10-Phenanthroline
Mn^{+2} (Quant strip)	Thiourea
Co^{+2} (Quant strip)	Phenylhydrazine hydrochloride
Ni^{+2} (Quant strip)	L-Ascorbic acid
Ag (Quant strip)	α-Furilmonoxime
s-Diphenylcarbazone[26]	Indicator paper pH 6.0-8.0 and $Na_2S_2O_3$
Alizarin red-S	Indicator paper pH 8.0-9.5 and $Na_2S_2O_3$
Xylenol orange	1,5-Diphenylcarbohydrazide
Dipycrylamine	Morin
2-Mercaptothioazoline	Pb-Diethyldithiocarbamate
Dithizone	Arsenic trisulfide
NaI (freshly prepared)	Rubeanic acid
Phenylhydrazine	PAN (1-(2-Pyridylazo)-2-naphthol)

From this initial screening, a selection of the most promising detector paper systems was made and evaluated more extensively with natural waters and soils. Results are presented in Table 4.

Extensive testing showed that the papers did indeed respond to chemical contamination. However, a certain variance was found in the reproducibility

TABLE 4. CANDIDATE DETECTOR PAPERS

Challenge Material	Chromate	Zn^{+2}	Ether Peroxide	Sulfite	Mn^{+2}	Ni^{+2}	Co^{+2}	Alizarin Red-S	4,4'Bio(demethylamino)-thiobenzophenone	PAN	PAR	Xylenol Orange	1,5-Diphenylcarboxhydrazide	s-Dipheryl carbazone	Thionalide	Dithizone	Phenylhydrazine hydrochloride	as-Diphenylhydrazine	Thiourea	Morin
Blank	ltP	O	C	C	C	W	C	T	Y	Y	Y	Y	ltP	ltP	C	BL	C	G	W	ltY
1. Antimony trioxide																				
2. Arsenic trisulfide																				
3. Cadmium chloride	Pk	R								BR-P	R			R		Y				
4. Lead thiocyanate	P	R						dkR		GR-P	R	P	Pk	R		R				
5. Mercuric cyanide		R														O				
6. Selenium dioxide		BR				O	O	Y							O	Y-GN	ltO		ltO	W
7. Zinc fluoride	R	R						Pk		P	R	dkR	Pk	R		R				
8. Sodium arsenate	Pk							P					ltPk	P		Y				
9. Sodium arsenite								P					Pk	Pk		Y				
10. Potassium bichromate	P	BR	GN-BL		BL	Y	Y						P					P		
11. Potassium chromate			GN-BL		BL	Y	Y	P					T	P		Y		P		
12. Potassium permanganate	BR	T	dkBL	R	BL	T		Pk	Pk	BR	R	T	BR	R		R			BR	

COLOR CODE: lt = light, dk = dark; P = purple, W = white, T = tan; BR = brown, BL = blue, O = orange; GN = green, C = cream; Y = yellow, GR = gray; R = red, Pk = pink

of results. The problem is largely associated with readout, which requires subjective evaluation of the presence and quality of certain colors. Very often, impregnated papers, whether prepared in-house or obtained commercially, have an initial color and it is up to the operator to determine whether there is a gain or a change in the hue of the original color. All of these situations were aggravated when testing was extended to natural waters and soils; the reproducibility became even more unreliable.

As a result, with the exception of several Quant strips which did prove to be reliable even under the most adverse circumstances, the detector paper approach was reduced in effort. As seen in the next section, the value of some of these reagents was exploited further by an alternate process.

Quant strips Mn^{+2}, ether peroxide, and Zn^{+2} were suitable for kit use. However, the Mn^{+2} and ether peroxide strips gave identical responses. Therefore, Mn^{+2} (either could be used) was selected. Zn^{+2} test strips became unavailable and therefore Ni^{+2} was selected as a backup test for selenium.

The findings are presented in Table 1.

INORGANICS SPRAY REAGENTS

Many of the reagents investigated in preparation of detector papers were examined again for use in spray form. It was believed that spray reagents, because they are applied fresh to the test spot and the surrounding area (which acts as a real-time reference blank), markedly improve the reproducibility of the tests. Because high reliability of identification is a primary goal in this program, the tests were judged to warrant the extra weight, volume, and cost required by the spray reagents.

Primary effort in this area was applied to the following reagent compositions:

1. Alizarin redS (alizarin sodium sulfonate), 0.05% in methanol
2. 4-2(2-Pyridylazo) resorcinol (PARK), 0.125% in methanol
3. Xylenol orange, 0.25% in methanol
4. s-Diphenylcarbazone, 0.5% in methanol
5. Diphenylthiocarbazone (dithizone), 0.05% in methanol

Tests were conducted by spotting 5-microliter aqueous samples (1 mg/ml) of contaminants on segments of silica gel HL Uniplates. The plates were dried in the oven or at room temperature and then sprayed with the candidate reagent. Color judgments were easier as the surrounding area acted as a reference blank.

As indicated in Thin-Layer Chromatography (TLC) earlier, these five reagents were packaged in self-contained pressurized spray cans. PAR (the can formulation is given) is one of the preparations that functions and can be stored satisfactorily. Through continued testing with natural water and soil samples, it was decided to include PAR, s-diphenylcarbazone, and diphenylthiocarbazone in the kit for the detection concept. Chromist sprayers are used in application of s-diphenylcarbazone and diphenylthiocarbazone. Details on use

of these reagents are given in the Appendix, Equipment and Methodology, and Analytical Procedures.

Findings are presented in Table 1.

RECOVERY AND CLEANUP

Tests for contamination in water using detector papers, CHEMets, inorganic reagent sprays, spot tests, arsine/Gutzeit, and detector tubes can be run directly on the water sample. Tests using TLC procedures all require that the contaminant be separated from water. No test was found for alcohol in water, nor was a means for recovering it from water found.

Primary effort for recovery of contaminants from water was placed on evaluation of the "Drug-Skreen" system[27] marketed by Brinkmann Instruments, Westbury, New York 11590. The system uses small prepackaged columns containing Amberlite XAD-2 resin, which is commonly used to extract many organic materials from aqueous solutions. Although the system is intended for recovery of abused drugs from urine samples, it does have a broader application.

The use of the "Drug-Skreen" system in the identification kit is shown in the Appendix, Equipment and Methodology. First, 20 ml of water sample containing a dissolved or suspended contaminant was passed through the column by gravity feed. Then, solvent was made to pass through the column and was saved in a collection flask. A siliconized phase filter was used to prevent residual water from entering the collection flask. Chloroform and methylene chloride were equally effective as recovery solvents. Ethyl acetate was not found to be a suitable solvent; it tended to form an emulsion with water, which tended to considerably slow down or altogether stop passage through the phase filter.

Selection of the solvent also had an influence on the subsequent TLC tests that could be used. For example, a number of reagents, such as indandione, fluorescein mercuric acetate, and 2', 7'-dichlorofluorescein, were dependent on a fluorescent readout. Fluorescent materials extracted from the Amberlite resin interfered with these procedures. Also, methylene chloride, which is a suitable solvent for alcohol-extracted material from the resin, interfered with the vanadium(V) 8-hydroxyquinoline test.

On the basis of these findings and the fact that chloroform is used in the kit for the TLC elutions, it was selected as the extraction solvent. Recovery of contaminants from water is shown in Table 5. It is seen that only acetic acid and maleic acid are not recovered from water using the Amberlite column and chloroform.

A limited investigation was made into the utility of using Conwed Sorbent Pads (Conwed Corporation, St. Paul, Minnesota 55101) as a means of recovering liquid contaminants. Conwed pads are made of highly absorptive materials and are used in cleanup of chemical contamination on the ground, other surfaces, and in water. The pads measure 45.7 cm x 45.7 cm x 0.64 cm and are largely composed of cellulose-type materials bonded to a polypropylene netting for strength.

TABLE 5. RECOVERY OF CONTAMINANTS FROM THE ENVIRONMENT

	Test Material	Water	Sand	Organic Soil
1.	Antimony trioxide	-	-	-
2.	Arsenic trisulfide	-	-	-
3.	Cadmium chloride	-	good (2)	good (2)
4.	Lead thiocyanate	-	good (2)	good (2)
5.	Mercuric cyanide	-	good (2)	good (2)
6.	Selenium dioxide	-	good (2)	good (2)
7.	Zinc fluoride	-	good (2)	good (2)
8.	Sodium arsenate	-	-	-
9.	Sodium arsenite	-	-	-
10.	Potassium chromate	-	good (2)	good (2)
11.	Potassium bichromate	-	good (2)	good (2)
12.	Potassium permanganate	-	good (2)	good (2)
13.	Propyl alcohol	NM	good (3)	good (3)
14.	Acetaldehyde	-	-	-
15.	Butylamine	good (1)	good (4)	poor (4)
16.	Triethylamine	good (1)	good (4)	poor (4)
17.	Aniline	good (1)	good (4)	good (4)
18.	Amyl acetate	-	-	-
19.	2,4-D (Esters)	good (1)	good (4)	good (4)
20.	Isoprene	-	-	-
21.	Naphthalene	-	-	-
22.	Acrylonitrile	-	-	-
23.	Benzonitrile	-	-	-
24.	Nitrophenol	good (1)	good (4)	good (4)
25.	Acetic Acid	poor (1)	good (4)	good (4)
26.	2,4-D (Acid)	good (1)	good (4)	good (4)
27.	Maleic acid	poor (1)	good (4)	poor (4)
28.	Benzoic acid	good (1)	good (4)	good (4)
29.	Dieldrin	good (1)	good (4)	good (4)
30.	Heptachlor	good (1)	good (4)	good (4)
31.	DDT	good (1)	good (4)	good (4)
32.	Chlorobenzene	-	-	-
33.	Diazinon	good (1)	good (4)	good (4)
34.	Malathion	good (1)	good (4)	good (4)
35.	Parathion	good (1)	good (4)	good (4)
36.	Phenol	good (1)	good (4)	good (4)

Recovery Method: - = Not applicable, NM = No method available, (1) = XAD-2 column with chloroform, (2) = Direct extraction with water, (3) = Direct extraction with chloroform, (4) = Direct extraction with methylene chloride.

Small samples of the pads were used to collect contaminants from water and soil and then extracted by chloroform or methylene chloride in preparation for analysis. However, blank tests showed very strong response with a large number of reagents, indicating that significant interfering materials were being extracted from the pads. In view of these findings and the fact that the pads are not intended nor quality-controlled for analytical work, the concept was abandoned.

For subsequent TLC tests, chloroform was used for extraction. For other tests, water was used for extraction. Methylene chloride is used as a collection solvent for alcohol in soil. As noted previously, chloroform cannot be used because it contains traces of alcohol. Unfortunately, a procedure for recovery of alcohol from water using methylene chloride was not developed. The method for recovery of contaminants from soil is given in the Appendix, Analytical Procedures.

The proposed procedures were evaluated using three types of soil--low organic (beach sand), medium organic (sand/clay mix, some organic), and high organic (dark soil, high organic content). Recovery of contaminants from soils having high or low organic content was essentially the same; therefore, these two soils were simply grouped together as organic soil. Findings are presented in Table 5. It is seen that recovery from sand was good in all cases. Recovery of contaminants from soils containing organic matter was generally good; significant exceptions were potassium permanganate, butylamine, triethylamine, and maleic acid.

EVALUATION IN THE NATURAL ENVIRONMENT

Tests detailed in the manual for the identification kit (Appendix) were evaluated on natural waters and soils. Water samples from Winter's Run, Otter Point Creek, and Big Gunpowder Falls, three fresh-water streams in Harford County, Maryland, were contaminated in the laboratory at concentrations of 1 mg contaminant/ml of water. Sandy, low organic, and high organic soils were collected from areas neighboring CSL and contaminated in the laboratory at approximate concentrations of 1 mg of contaminant per loosely packed ml of soil.

All contaminants were detected with the test procedures with the exception of those in Table 5 that were marked as poorly recovered. Findings with waterborne contaminants were generally more reliable. Response with TLC, detector tubes, detector papers, arsine/Gutzeit, spot tests, and CHEMets was good. The greatest variation was found with the inorganic spray reagents. The reactions were time-dependent and subject to a number of other factors, such as degree of dryness of the plates, temperature, etc. As a result, variations were found in colors and in the minimum detection sensitivity. However, the degree of variation was not so great as to preclude use of these reagents, which contributed much valuable information toward identification of inorganic contaminants.

It is evident that testing in soil is a nonroutine, highly complex job requiring great deductive reasoning and skill on the part of the operator. Soil presents a very complex background from which individual pieces of data must be extracted and then presented as a meaningful assemblage of information. The complexity of the problem is evidenced by the chloroform extracts of the soil

samples, which can range from colorless for beach sand to dark brown for some of the soils containing a high organic content. While the effects of background for the sand samples are minimal, soils with a high organic matter content often show a significant loss of sensitivity. However, in the case of a very sensitive test, such as the hydrogen peroxide CHEMet, care must be taken even with sandy soils.

Loss of response may be attributed to interactions between the contaminant and/or reagent with the soil background. This can be a particular problem with acid/base compounds in soils of different acidity. Another example of loss of response is demonstrated by potassium permanganate; it is easily identified in sandy soils but not in those containing organic matter. The permanganate is reduced by organics and is no longer detected by the proposed procedures. The reaction of the permanganate ion with the soil background is evident from the extracts, wherein those from sand are colored purple, and those from organic soils are light yellow.

It is possible to identify some contaminants by their decomposition products. However, the processes are very complex and were not within the scope of this program. It may be concluded that the probability of detecting and identifying both inorganic and organic contamination in loose beach sand is very good. Identification in soil with high organic content is also possible in some cases, but one should proceed with greater caution.

As part of this program, an interference study was conducted to determine the proposed kit's ability to function in the natural environment. A random collection of water and soil samples was made from a circuitous route about the upper portion of the Chesapeake Bay in Maryland extending down to the William Preston Lane, Jr. Memorial Bridge. Water samples were taken from fresh-water streams that drain into the bay; soil samples were taken from locales adjacent to the water sources. Assuming that the samples collected were free of chemical contamination, the number and types of interferences were noted.

As has been indicated, interference from constituents normally found in inland waters is negligible with the proposed procedures. However, as has also been indicated, extracts of soils with high organic content are often dark and contain numerous potential interferences. The extracts of many of the soils used in this study were extremely dark, although a few were nearly colorless.

On the basis of data presented in Table 6, one responding to a spill incident might suspect contamination in soil samples 8 and 15. Such information in combination with the circumstances surrounding that particular locale might be the basis of followup analysis at a laboratory. On the other hand, if there are no mitigating circumstances to support the findings, one would proceed with more caution. Those responses noted in the table that are consistently weak are probably attributable to low-level background constituents.

Accordingly, it is recommended in the manual that sampling should be accomplished in the following order of preference: (1) neat materials, (2) water samples, (3) sand/clay-type soil samples, and (4) dark, high organic soils (very undesirable).

TABLE 6. INTERFERENCE SCREENING OF KIT PROCEDURES WITH RANDOM WATER AND SOIL SAMPLES

Water Samples	Mn^{++}	Ni^{++}	H_2O_2	Phenols	PAR	s-Diphenyl carbazone	Dithizone	Nitro Compounds	Aldehydes	Esters	Arsine/ Gutzeit	Benzene Draeger	Olefin Draeger	Benzene Ben/Gas	Acetone Ben/Gas	Bromocresol G	Bromocresol G + UV	$AgNO_3$ + UV	Chloranil + heat	$PdCl_2$	Alcohols
1. East Creek, Rte 40																					
2. Creek North of Aberdeen, Rte 40																					
3. Severn River, Rte 40																					
4. Kentnarrows Bridge Rtes 301 & 50																					
5. Stream North of Sudlersville																					
6. Stream, Centreville, Rte 213																					
7. Stream, North of Centreville, Rte 213																					
8. Stream, Galena, Rte 213 & Davis Rd																					
9. Marsh Stream, Chestertown																					
10. Chester Stream, Chester																					
11. Bohemia River, Chestertown																					
12. C&D Canal, N. Chesapeake City																					
13. Farr Creek, Elkton, Rte 7																					
14. Williston Lake, Denton																					
15. Choptank River, Greensboro																					
Soil Samples																					
1. East Creek, Rte 40			±			±															
2. Creek North of Aberdeen, Rte 40						±															
3. Severn River, Rte 40										±											
4. Kentnarrows Bridge Rtes 301 & 50																					
5. Stream North of Sudlersville																					
6. Stream, Centreville Rte 213			±																±		
7. Stream, North of Centreville, Rte 213																					
8. Stream, Galena, Rte 213 & Davis Rd																		+	+	±	
9. Marsh Stream, Chestertown																					
10. Chester River, Chestertown					±	±							±						±	±	
11. Bohemia River, Chestertown																					
12. C&D Canal, N. Chesapeake City																					
13. Farr Creek, Elkton, Rte 7																					
14. Williston Lake, Denton																					
15. Choptank River, Greensboro																		+	±	±	

NOTE: + = Strong Test; ± = Weak Test

STORAGE

Reagents that showed potential for use in the kit were prepared and placed in storage to obtain an estimate of shelf life. This evaluation included only reagents prepared on this program, not commercially available spray reagents, detector papers, or detector tubes.

Except as noted in Table 7, the reagents were kept in a bin in tightly stoppered but unsealed brown bottles. The reagents in bottles would be similarly supplied in the kit but provided with Chromist spray assemblies for attachment when needed. The reagents were tested at monthly intervals. Their useful life is noted.

It was noted that some sediment began forming in some of the bottled reagents after 3 to 4 months, but this did not interfere with the reagent's ability to respond to the test materials.

Reagents for the various spot tests were also placed in storage. However, since many of these were still being modified up to several months ago, data on them is not as extensive. Nonetheless, they are still reactive after 2 months and should continue to be so for many more.

TABLE 7. SHELF STORAGE OF CHEMICAL REAGENTS

Reagent	Useful Life
1. PAR, 0.16 gm/250 ml methanol	About 6 months
2. s-Diphenylcarbazone, 1.25 gm/250 ml chloroform	About 6 months
3. Dithi Ver™, 10 capsules/250 ml chloroform	About 6 months
4. Dithizone, 0.125 gm/250 ml chloroform	About 6 months
5. Silver nitrate, 1.25 gm/250 ml water	About 6 months
6. Chloranil, 1.25 gm/250 ml chloroform	About 6 months
7. Palladium chloride, 1.25 gm/250 ml methanol	About 6 months
8. PAR, pressurized can	Over 6 months
9. Chloranil, pressurized can	Over 6 months

SECTION 5

IDENTIFICATION CONCEPT

It is obvious that a kit that attempts to identify 300 materials would require a considerable number of tests. It would not be practical, even if the tests were available (which they were not), to include a test for each compound. Accordingly, it was decided to select a group of tests that had a range of response, which, used in combination, would serve to specify certain materials or classes. Therefore, an attempt was made to select from the tests studied in this program a group that was neither too narrow nor too general in response. These tests are specified in the manual (Appendix). The purpose of selecting these tests was not only to identify the 36 test materials studied in this program, but also to provide a range of response that may be capable of identifying other materials from the Federal list of hazardous substances.

Documenting the response data with only the 36 compounds used in this test program was relatively easy. However, if additional test data were produced with other materials, classification and retrieval would have become increasingly difficult. Accordingly, a simple, manually operated retrieval system was designed based on the use of Instant Data System (IDS) cards (Professional Aids Co., Chicago, Illinois 60606). The identification kits each have a set of 36 cards--one for each of the test materials studied.

The IDA cards were printed with a standard format and pertinent data, such as material description, response to different tests, R_f and R_S values, and graphic representations of TLC separations. The cards were notched at appropriate positions to correspond to the test data. A card prepared for the identification of malathion is shown in the Appendix, Equipment and Methodology.

If it is decided to screen other materials through the kit procedures, similar cards can be prepared and added to the data base. If the collection of cards becomes unwieldy, sorting needles may then be used to facilitate separations.

In addition to the above procedures, the identification kit has been cross-referenced with EPA's detector kit to obtain additional identification capability (Appendix, Supplementary Identification with the Hazardous Materials Detector Kit).

Information entered on the IDS cards for the materials studied in this program is noted below. The R_f and R_S data is based on separations made with chloroform in a sandwich chamber at room temperature (see Experimental, Thin-Layer Chromatography, and also the Appendix, Equipment and Methodology).

Consideration was given to indexing the R_f and R_s values on the IDS cards. However, it was decided that even with use of a reference dye, values were subject to significant variation, particularly due to temperature. Accordingly, it was decided not to notch the values, but to include them on the cards along with the graphic portrayal of TLC separations, as qualitative information.

Numbers associated with test descriptors refer to analytical procedures given in the manual (Appendix, Analytical Procedures).

DATA ENTERED ON IDS CARDS FOR 36 HAZARDOUS MATERIALS

1. ANTIMONY TRIOXIDE, colorless crystalline powder, slightly soluble in water.
   11. gray-blue

2. ARSENIC TRISULFIDE, yellow crystals or powder, insoluble in water.
   11. gray-blue

3. CADMIUM CHLORIDE, small white crystals, soluble in water.
   3. light yellow precipitate
   4. light white precipitate
   5. red-purple
   6. red-purple
   7. yellow

4. LEAD THIOCYANATE, white or light yellow crystals, slightly soluble in cold water.
   3. light black precipitate
   4. light white precipitate
   5. red-purple
   6. red-purple
   7. orange

5. MERCURIC CYANIDE, colorless crystals, slightly soluble in water.
   3. light brown
   5. weak purple
   6. weak purple
   7. yellow

6. SELENIUM OXIDE, colorless needles, soluble in water.
   2. orange
   3. light yellow
   5. weak yellow-orange
   7. yellow-green

7. ZINC FLUORIDE, white powder, slightly soluble in water.
   4. light yellow-green precipitate
   5. red-purple
   6. bright red-purple
   7. red-orange

8. SODIUM ARSENATE, colorless crystals, soluble in water.
   7. small yellow spot
   11. gray-blue

9. SODIUM ARSENITE, grayish-white powder, soluble in water.
   3. light yellow
   11. gray-blue

10. POTASSIUM BICHROMATE, yellowish-red crystals, soluble in water.
   1. dark green
   3. dark brown
   5. weak purple
   6. purple
   7. yellow

11. POTASSIUM CHROMATE, yellowish-red crystals, soluble in water.
   1. dark green
   3. dark brown
   5. weak purple
   6. purple
   7. yellow

12. POTASSIUM PERMANGANATE, dark violet crystals, soluble in water.
   1. dark green
   3. dark brown
   4. light yellow-brown precipitate
   5. purple
   6. red-purple
   7. gray

13. PROPYL ALCOHOL, colorless liquid, soluble in water.
   3. light brown
   21. red; test is only applicable to neat samples and soil testing

14. ACETALDEHYDE, colorless liquid, miscible with water.
   9. purple
   13. light brown
   15. green

15. BUTYLAMINE
   3. light brown
   16. blue
   18. dark brown to black
   19. purple
   20. white
   $R_f = 0.0$, $R_s = 0.0$

16. TRIETHYLAMINE, colorless liquid, slightly soluble in water.
   3. light brown color
   16. blue
   18. gray

19. purple
20. light brown
$R_f = 0.0$, $R_s = 0.0$

17. ANILINE, colorless liquid, slightly soluble in water.
4. brown
12. purple on precleanse layer, purple on indicator layer
18. dark brown to black
19. yellow-green
20. light brown
$R_f = 0.33$, $R_s = 0.46$

18. AMYL ACETATE, colorless liquid, very slightly soluble in water.
10. purple
15. black

19. 2,4-D (ESTERS), colorless liquid, very slightly soluble in water.
10. brown-orange
17. light yellow
18. gray
$R_f = 0.70$, $R_s = 0.98$

20. ISOPRENE, colorless volatile liquid, insoluble in water.
12. dark brown on precleanse layer
13. light brown
14. dark yellow
15. dark brown

21. NAPHTHALENE, white crystalline solid, insoluble in water.
12. dark blue on precleanse layer
14. dark gray

22. ACRYLONITRILE, colorless liquid, partially miscible in water.
13. light brown
15. brown-green

23. BENZONITRILE, colorless liquid, slightly soluble in water.

24. NITROPHENOL, yellow crystals, soluble in water.
4. red-orange
8. dark yellow
$R_f = 0.83$, $R_s = 1.16$

25. ACETIC ACID, colorless liquid, miscible in water.
16. weak yellow
18. light brown-black
$R_f = 0.07$, $R_s = 0.10$

26. 2,4-D (ACID), white to yellow crystalline solid, slightly soluble in water.
12. red-brown on precleanse layer
15. yellow-black

16. yellow
18. brown to black
19. light white
20. light white
$R_f = 0.09$, $R_s = 0.12$

27. MALEIC ACID, colorless crystals, soluble in water.
16. yellow
18. white
19. light white
20. light white
$R_f = 0.0$, $R_s = 0.0$

28. BENZOIC ACID, colorless to white needles or scales, very slightly soluble in water.
16. yellow
18. white to light gray
19. light white
20. light white
$R_f = 0.13$, $R_s = 0.18$

29. DIELDRIN, pale tan flakes, insoluble in water.
17. yellow
18. gray to white with brown ring
$R_f = 0.90$, $R_s = 1.26$

30. HEPTACHLOR, white waxy solid, insoluble in water.
17. yellow
18. gray to white with brown ring
$R_f = 1.00$, $R_s = 1.40$

31. DDT, colorless or white powder, insoluble in water.
17. yellow
18. gray to white with brown ring
$R_f = 1.00$, $R_s = 1.40$

32. CHLOROBENZENE, colorless volatile liquid, insoluble in water.
12. light red on indicating layer
14. gray
15. dark brown

33. DIAZINON, colorless liquid, slightly soluble in water.
12. red-purple on precleanse layer, red-purple on indicating layer
14. gray-green
15. brown-green
18. white to white with gray ring
20. brown to yellow with brown ring
$R_f = 0.27$, $R_s = 0.38$

34. MALATHION, yellow to dark brown liquid, very slightly soluble in water.
14. gray-green

15. dark green
18. white to white with gray ring
20. brown to yellow with brown ring
$R_f = 0.33$, $R_s = 0.46$

35. PARATHION, yellow liquid, slightly soluble in water.
3. light brown
14. yellow-green
15. green-yellow
18. white to white with gray ring
20. brown to yellow with brown ring
$R_f = 0.76$, $R_s = 1.06$

36. PHENOL, white crystalline mass, soluble in water.
4. dark purple
18. gray-brown
19. light orange
$R_f = 0.16$, $R_s = 0.22$

SECTION 6

PROTOTYPE KITS

This program has resulted in a two-component identification kit--an inverter/shortwave UV lamp unit for photochemical reactions and a larger reagent/auxiliary equipment package. Prototype kits fabricated according to this design are shown in Figures 5, 6, and 7. The manual for the kit's use is given in the Appendix of this report. Two prototype kits, drawings, and parts lists were provided to EPA under separate cover.

The inverter/UV lamp unit measures about 7 in x 8 in x 16 in (17.8 x 20.3 x 40.6 cm) and weighs 15 lb (6.8 kg). The reagents/equipment package measures 8 in x 10 in x 24 in (20.3 x 25.4 x 61.0 cm) and fully packed, weighs 13.5 kg (30 lb). The procedures include thin-layer chromatography, commercial detector tubes, spot tests, an arsine/Gutzeit test, CHEMets, detector papers, a variety of spray reagents, and cleanup and recovery equipment.

The photochemical tests required the use of a high-intensity shortwave UV light. In reviewing the availability of high-intensity lamps, none were found that are battery-powered. Accordingly, for use in the field, it was decided to use an inverter to drive the lamp by converting power from the transporting vehicle's 12-V DC battery to 115-V AC. Two reasonably compact inverters were ordered from Trippe Manufacturing Company, Chicago, Illinois 60606. Model PV-115 was found to deliver insufficient power, but Model PV-200 was adequate to develop the photochemical reactions.

Use of the inverter significantly added to the size of the kit. It measures 5 in x 5 in x 3 in (12.7 x 12.7 x 7.6 cm) and weighs about 6 lb (2.7 kg). However, the inverter was considered critical to the kit concept and was accepted nonetheless. Furthermore, it was found that the inverter emits a considerable amount of heat. Accordingly, the cooling fins were modified to function as a small low-temperature hotplate, which is used to develop the chloranil test.

The UV lamp used with the inverter is Mineralight, Model R-52, from Ultra-Violet Products, Inc., San Gabriel, California 91778. In operation with a standard-size vehicle, the UV lamp/inverter unit drew 7.2 amps from a 12-V DC battery with the engine off/ with the engine in idle, it drew 7.5 amps. It is planned to run the photochemical tests and the chloranil test together for a 10-minute period. There is no problem expected in using the UV lamp/inverter unit in this mode. The unit has a cable for hooking directly to the vehicular battery.

Figure 5. Hazardous materials spills identification kit, inverter/UV lamp unit.

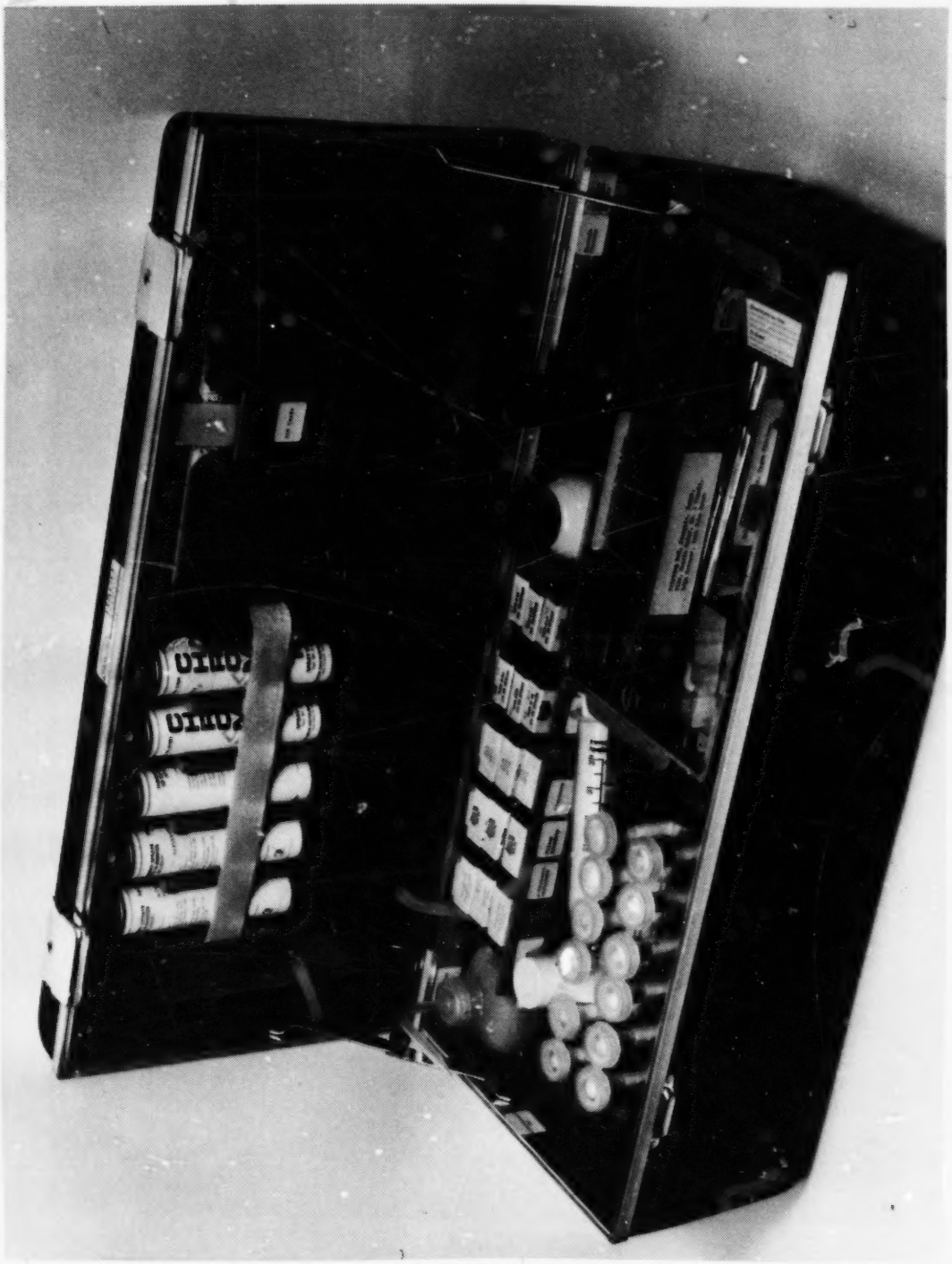

Figure 6. Hazardous materials spills identification kit, reagents/auxiliary equipment package.

Figure 7. Hazardous materials spills identification kit, reagents/auxiliary equipment package.

Many of the other components used in the identification kit are commercially available. However, a wide range of reagents were developed especially for its use. Unfortunately, some of them have limited shelf life and, accordingly, should be prepared on a regular basis or they should be monitored. More information on stability has been given in Section 4, Experimental Results.

A loading diagram for the kit is shown in the Appendix, Equipment and Methodology.

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LIST OF MANUFACTURERS AND SUPPLIERS

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2. Bendix/Gastec: National Environmental Instruments, Inc., Warwick, Rhode Island 02888
3. Brinkmann Instruments, Inc., Westbury, New York 11590
4. Case-Mason Filling, Inc., Joppa, Maryland 21085
5. CHEMetrics, Inc., Warrenton, Virginia 22186
6. Conwed Corporation, St. Paul, Minnesota 55101
7. Draeger: National Mine Service Co., Pittsburgh, Pennsylvania 15219
8. Fisher Scientific Co., Silver Spring, Maryland 20910
9. Gelman Instrument Co., Ann Arbor, Michigan 48106
10. Kitagawa: Matheson Gas Products, Lyndhurst, New Jersey 07071
11. Midwest Research Institute, Kansas City, Missouri 64110
12. Mine Safety Appliances, Pittsburgh, Pennsylvania 15235
13. Professional Aids Co., Chicago, Illinois 60606
14. Schleicher and Schuell, Inc., Keene, New Hampshire 03431
15. Scientific Products, Division of American Hospital Supply Corp., McGraw, Illinois 60085
16. Trippe Manufacturing Co., Chicago, Illinois 60606
17. VWR Scientific, Inc., Baltimore, Maryland 21227

APPENDIX

OPERATOR'S MANUAL FOR HAZARDOUS MATERIALS IDENTIFICATION KIT

SECTION 1

INTRODUCTION

The Hazardous Materials Spills Identification Kit (ID Kit) is intended to aid in the identification of hazardous materials spills in water and soils. The kit consists of two units -- an inverter/shortwave UV lamp unit for photochemical reactions, and a larger package with reagents and accessory equipment.

The ID Kit uses a variety of commercial and laboratory tests, which are adapted for field use. Because many tests are not "shelf-available," it is necessary for the operator to maintain the reagents in a ready-state condition. This may require monitoring and preparation of fresh batches on a regular basis.

Included in the ID Kit are test papers, detector tubes, spot tests, spray reagents, and thin-layer chromatography (TLC). In addition, there is equipment to facilitate the recovery of contaminants from water and soil.

The skills required to operate the kit are easily learned; however, the task of interpreting the derived data is more challenging. It is evident that the operator's level of training and background experience will play a significant role in combining and interpreting certain pieces of information in order to come to reasonable conclusions.

Identification may be accomplished by comparing the data derived from testing with that contained in the data system included with the kit. By comparing vital pieces of information, it is possible to identify some materials specifically, or in other cases, narrow them to particular classes.

It will be found that a number of factors affect the performance of the kit. For example, when the kit was developed, testing was accomplished using relatively pure test materials. In a "real-world" situation the time between the occurrence of a spill and its analysis will have a significant effect on oxidation, hydrolysis, and decomposition of the contaminant. The results can be shifts in color response, multiple TLC spots, and loss of sensitivity. It is evident that analysis should commence at the site as quickly as possible and with neat material -- solid or liquid -- if possible. If neat materials are not present, then the order in which samples should be taken are: (1) water, (2) clean sandy soil, (3) sand-clay mix, and (4) black, loamy soil. Interfer-

ence, in the last case, is significant and should be avoided when possible.

The ID Kit is largely designed to function alone. However, if the EPA's Detection Kit for Spilled Hazardous Materials is available, additional information can be gained, particularly in regard to inorganic contamination. Accordingly, a section of this manual describes supplementary identifications, which can be accomplished using the Detection Kit.

SECTION 2

EQUIPMENT AND METHODOLOGY

The components of the ID Kit are shown in Figure A-1 (the inverter/lamp unit) and Figure A-2 (the reagents and equipment loading diagram). The test procedures used in the kit are described in the analytical section; specialized processes or procedures are presented in the following paragraphs.

Sampling and Recovery. The contaminant at the spill site may be found under a variety of conditions. That is, it may be suspended or dissolved in water, intimately mixed with the soil, or neat, as a pile of dry material or a puddle of liquid.

Water samples to be analyzed by TLC procedures require that the contaminant be extracted in an organic solvent. The apparatus shown in Figure A-3 is used to accomplish this task.

Remove the upper sealing cap and snip off the sealing tip from a column. Press-fit a reservoir cup to the top of the column and suspend as shown from the side of the case. Pass 20 ml of water containing dissolved or suspended contaminants through the column. (Swirl the sample to obtain a representative sample of suspended matter.) Water that passes through the column may be discarded. Excess water may be forced through by applying pressure to the reservoir cup with the palm of the hand.

Press-fit a filter cartridge to the lower end of the column. Add 10 ml of chloroform to the reservoir and loosen the upper cotton plug with tweezers. The filter cartridge contains a phase filter, which allows the solvent to pass through while retaining excess water. Collect the chloroform extract in the small bottle and use it for the TLC tests.

In many instances described in the analytical procedures, it is necessary to make an extraction of the soil sample. Use the filtering apparatus described below to filter out all suspended matter prior to analysis.

When neat samples of contaminants are encountered, they may be dissolved directly in water or chloroform as specified in the test procedures.

Filter Assembly. It may be desirable in some instances to filter the extract from soil samples. For this purpose, the apparatus shown in Figure A-4, for which glass fiber filters are provided, is used to obtain clear samples. A sample of the extract is drawn into the syringe; a filter holder with a filter in place is press-fitted to the tip of the syringe; and a sample of extract is forced thorugh the Tygon tube into a 50-ml beaker for analysis.

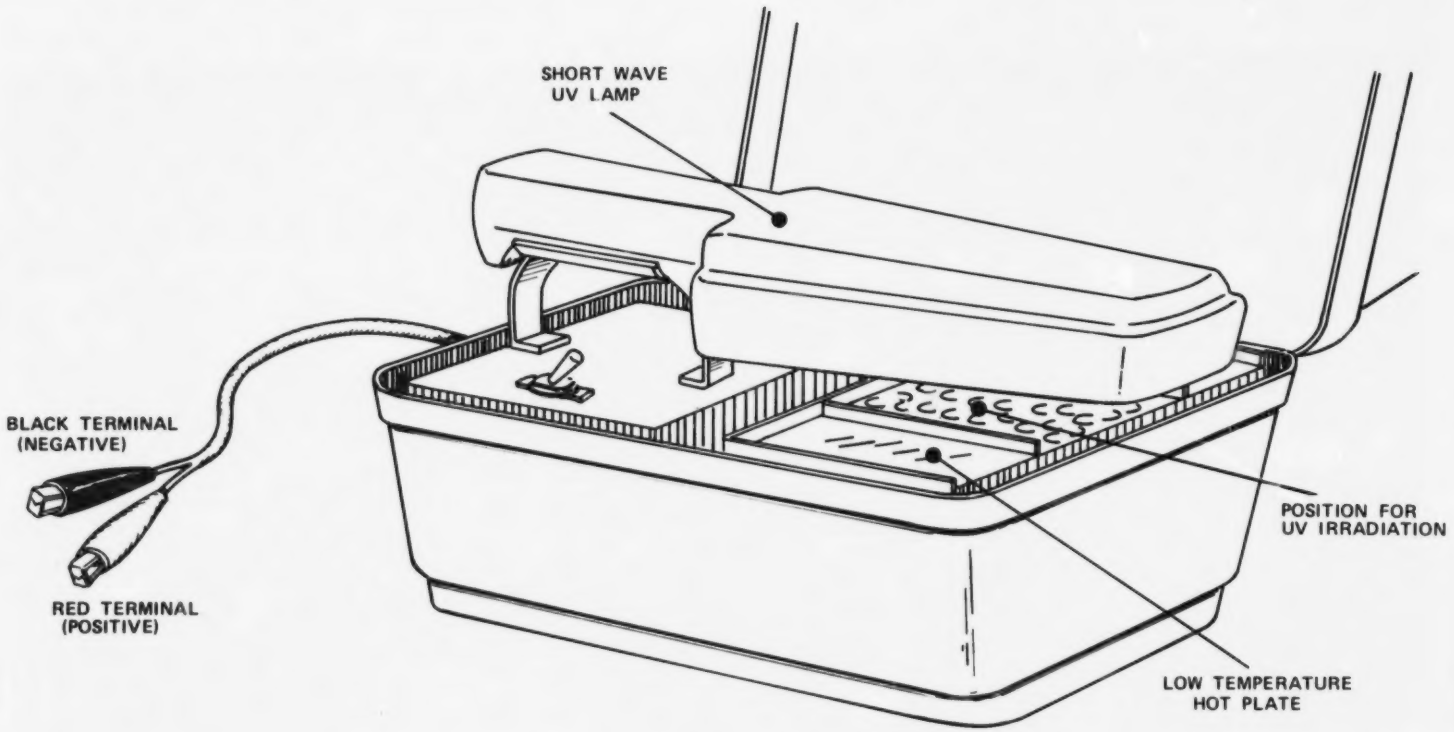

Figure A-1. Inverter/lamp unit.

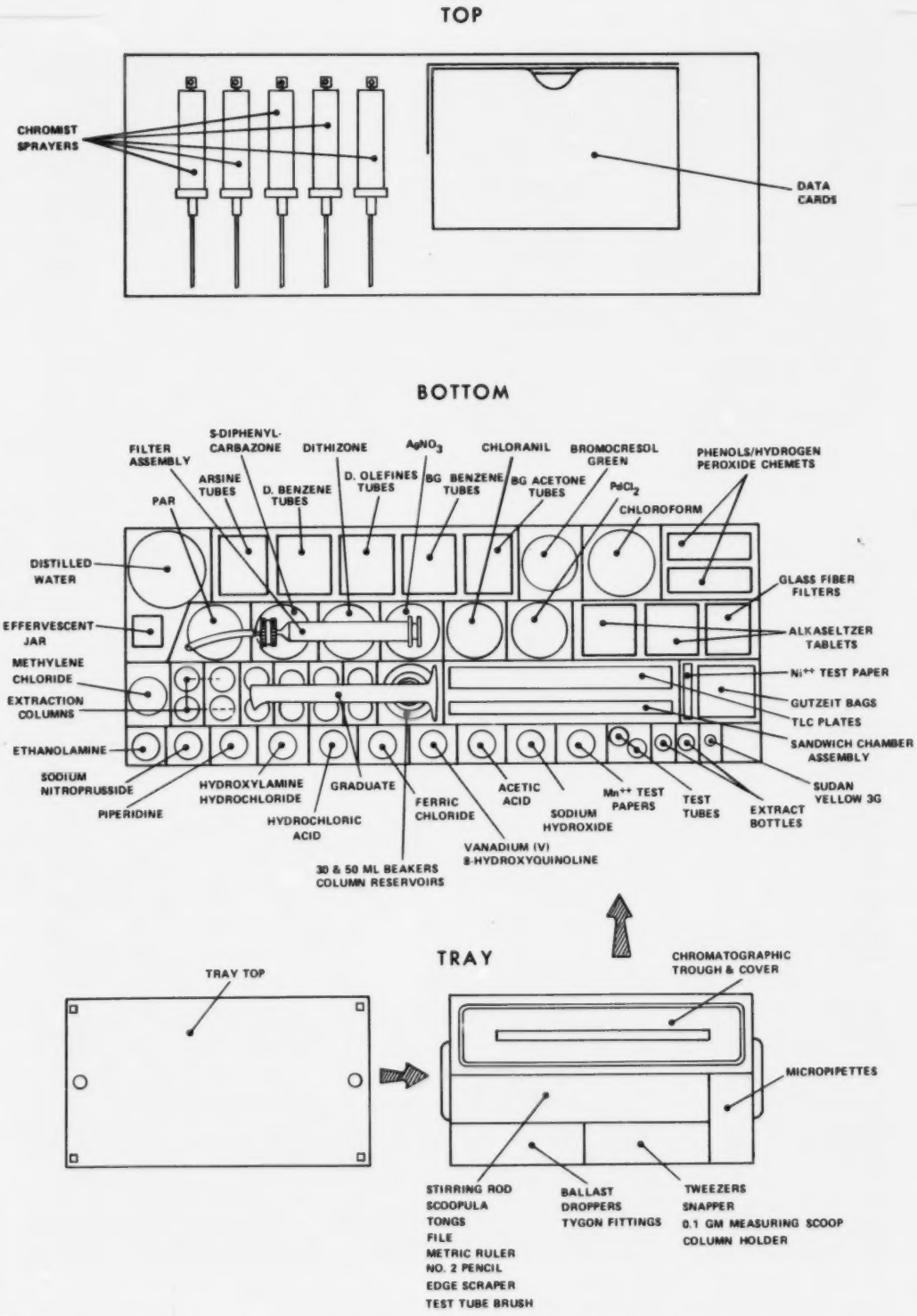

Figure A-2. Reagents and equipment loading diagram.

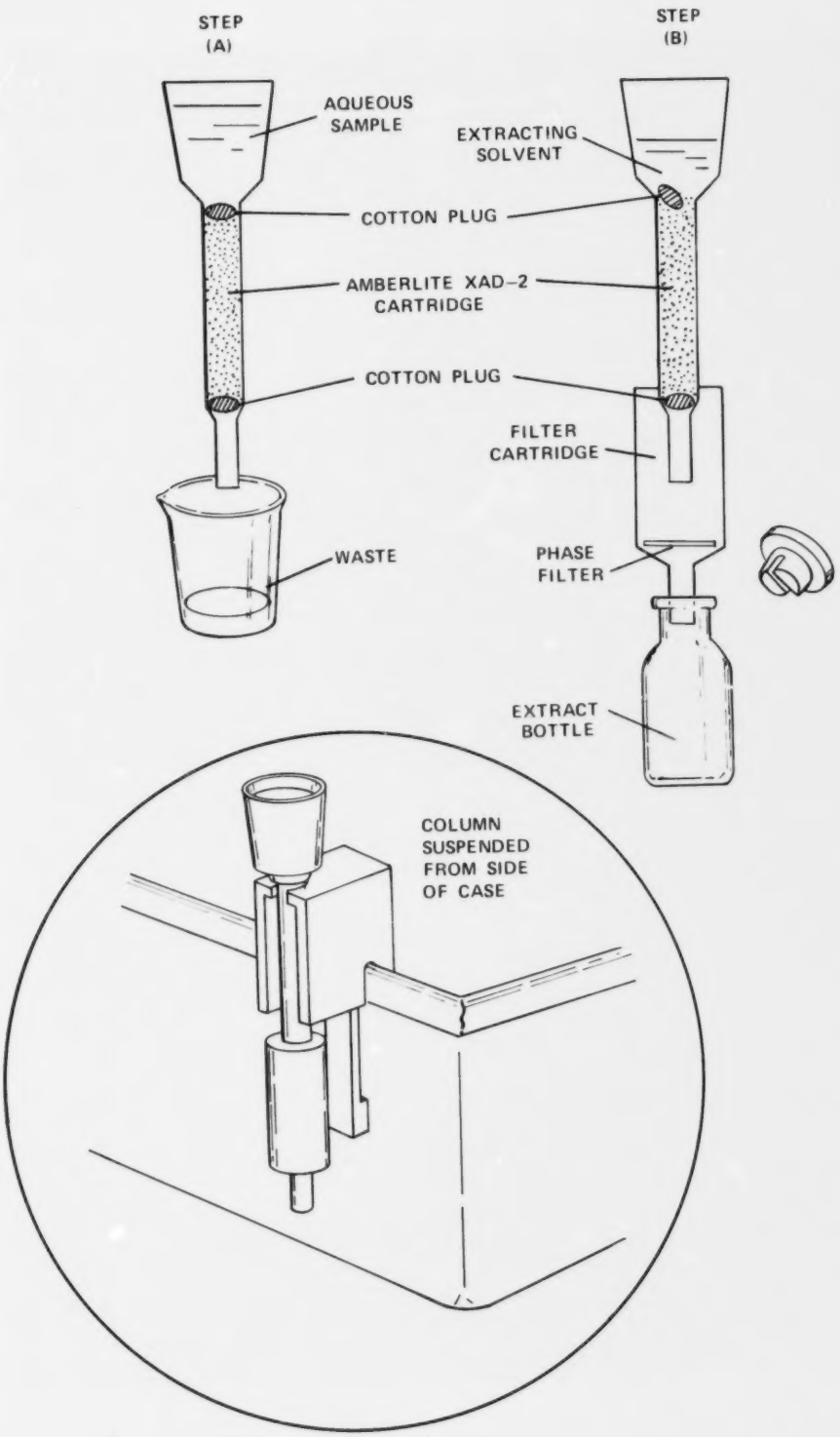

Figure A-3. Extraction system for water.

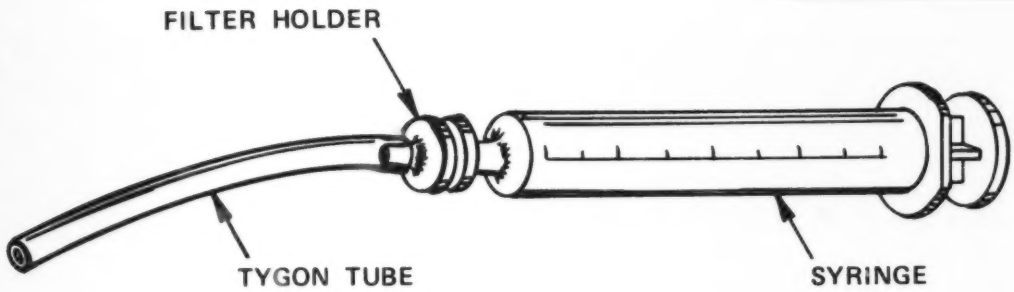

Figure 4. Filter assembly.

Inverter/Lamp Unit. The high-intensity shortwave UV lamp is required for photochemical reactions. The lamp operates on 110-V AC, which is provided by the inverter. The inverter, in turn, derives power from a common 12-V DC battery, such as is found in the transporting vehicle.

The unit is attached to the vehicular battery with provided clips and cable. Pay particular attention when attaching the positive (red) and negative (black) clips to the proper poles of the battery. Once hookup is made, both the lamp and inverter switches should be in the off position until needed. It should be noted that the inverter, when on, will draw current even if the lamp is off.

The inverter, which emits considerable heat, has been modified to act as a low-temperature hot plate for several tests. The positioning of TLC plates for heating and shortwave irradiation is shown in Figure A-1. The TLC plates are most easily positioned by removing the lamp from its retaining handle.

The inverter/lamp unit can be used to accomplish the required tests in about 10 minutes. In order to minimize battery drain, it is recommended that the tests be planned to run simultaneously. If the vehicular battery is in good condition, the power drain is insignificant. If there is some question, the inverter/lamp unit can be functioning while the transporting vehicle is idling.

Be certain that both the inverter and lamp switches are turned off when tests are completed.

CHEMets. CHEMets are glass ampoules containing color-forming reagents under vacuum; some ampoules have additional reagents coated on the surface of the tapered tip. To conduct a test with a CHEMet, collect a sample of water in a beaker. Place a CHEMet in the barrel of the Snapper, tapered tip first (Figure A-5). Immerse the end of the Snapper below the water level and press the CHEMet with your thumb to break the tip. The ampoule will fill with water; invert several times to assure complete mixing. Note the color and/or precipitate that may form.

Detector Tubes. Some contaminants can be expelled from water by an effervescent material and, thereby, detected by detector tubes normally used for

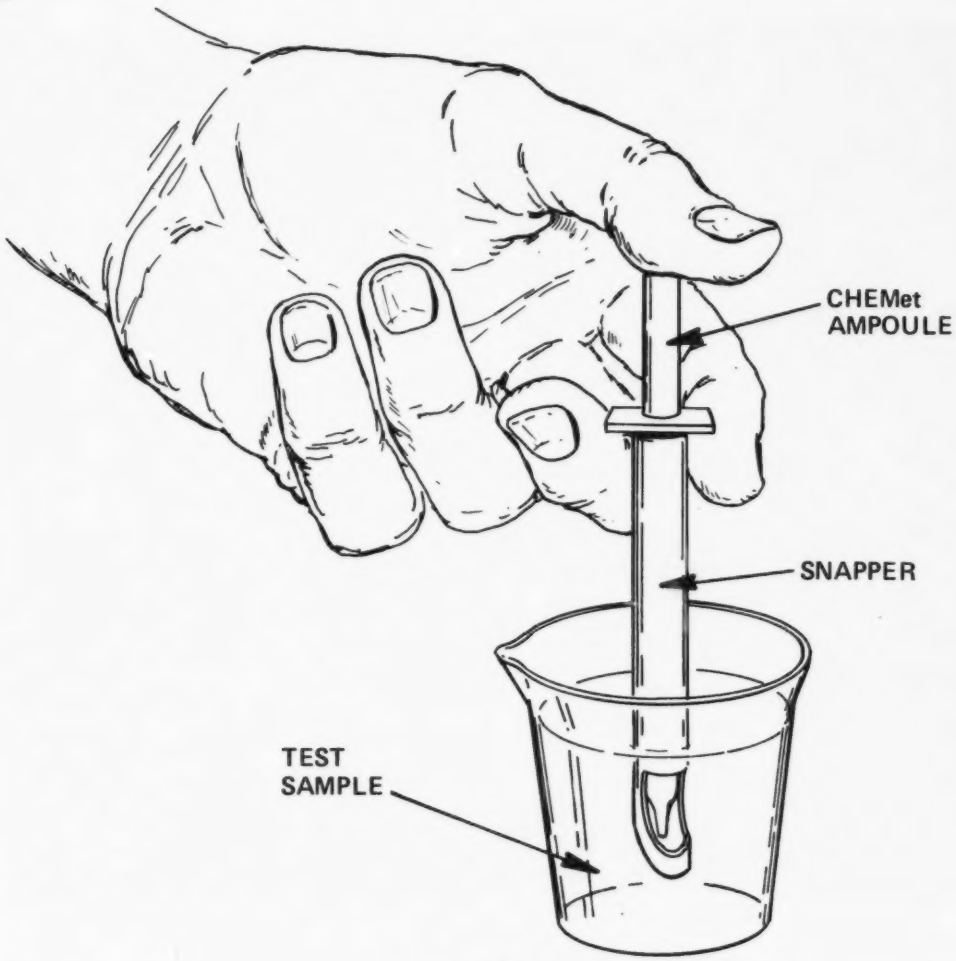

Figure A-5. CHEMets.

airborne contaminants. The test is performed using the assembly shown in Figure A-6. Depending on the test being conducted, effervescence can be provided using Alka-Seltzer^{R} tablets or a zinc/potassium bisulfate/cupric sulfate mix.

The test is performed with 80 ml of water in a jar. A file is used to score and remove the tips of a detector tube. The tube is inserted in the stopper top with the arrows pointing away from the jar. Several sizes of Tygon tubing are provided to accommodate the different size detector tubes.

The effervescent material is dropped in the water and the stopper quickly forced into the jar. The Draeger tubes have little resistance and the top need be held only lightly in place. The Bendix/Gastec tubes require that the stopper be held firmly. Caution should be taken to preclude water from entering the tubes, since it may interfere with the reaction. When effervescence

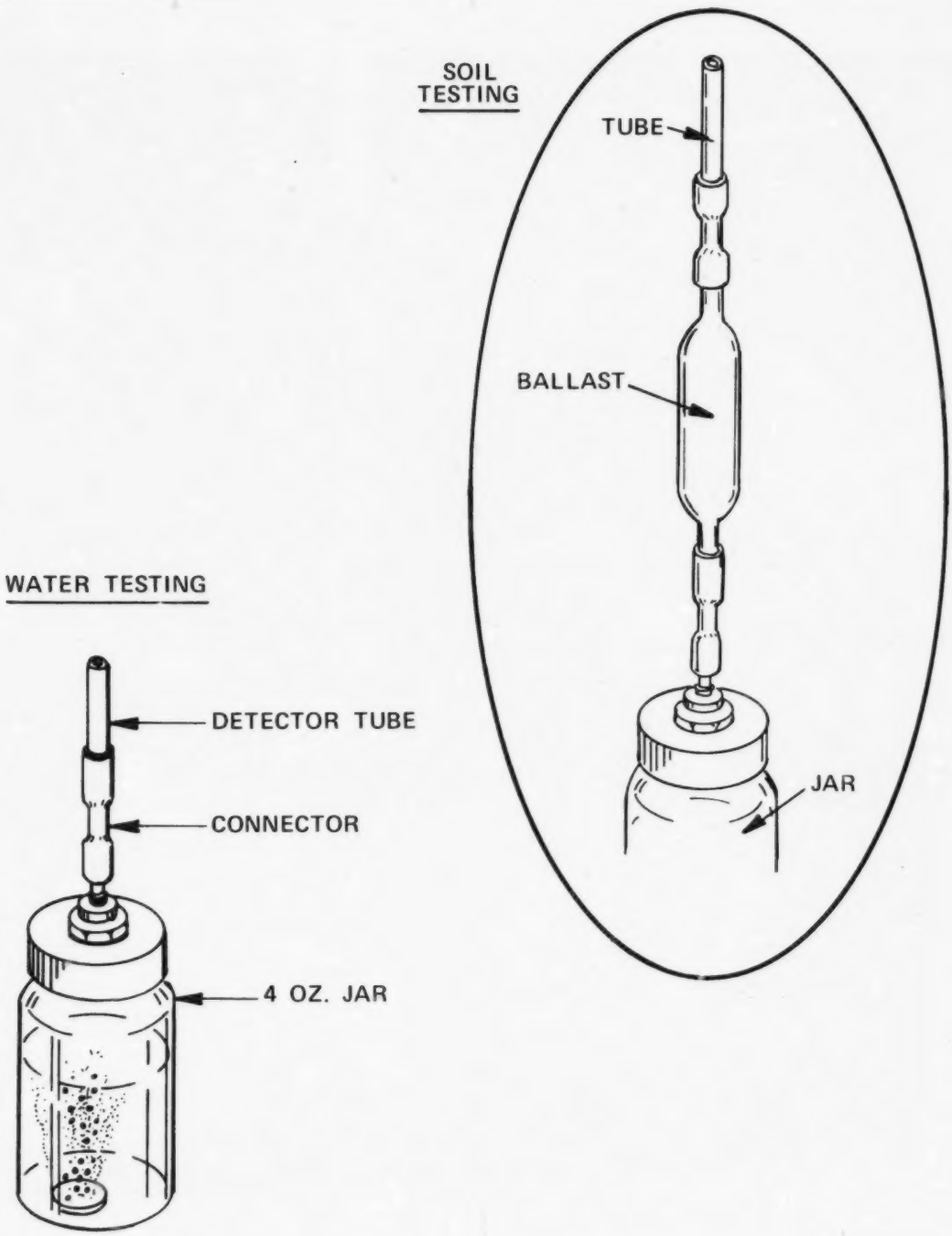

Figure A-6. Effervescent jar assembly.

is complete, color forming on any part of the tubes is noted.

It is found that when this apparatus is used to test for contamination in soil, considerable foaming can occur, particularly with soils having a high organic content. To preclude water from entering the tubes, a ballast tank (Figure A-6) is inserted in the system between the tubes and the stopper top.

Spray Reagents. Several of the reagents used in the kit are best applied in a spray form. Two of them, PAR and Bromocresol Green, are self-contained and ready for use as received.

The other reagents require that the propellant and the reagent be packaged separately. When they are needed, the Chromist Spray Unit is screwed onto the reagent reservoir and applied in the conventional manner. To avoid cross-contamination, the spray units should not be interchanged with the reagents. To extend the life of the reagents, they should be resealed as soon as possible with the original lids.

Thin-Layer Chromatography. The TLC procedures are the most complex and sophisticated portion of the ID Kit. At the same time, they can provide some of the most useful data to effect identification.

Prepare a TLC plate as shown in Figure A-7. Using a ruler, draw a light line with a soft lead pencil 10 mm from the bottom of the plate; designate this the origin. Try not to damage the surface of the silica gel. Draw another line 80 mm from the bottom of the plate and designate it as the solvent front. With the scraper, remove a 15-mm width of silica gel from each end of the plate.

Spot the plate as follows: Ignore the first segment; spot 8 microliters of chloroform extract in the center of each of the 2nd, 3rd, 4th, 5th, and 6th segments on the pencil line that represents the origin. On the 7th segment, spot 3 to 4 microliters (estimated) of Sudan Yellow 3G standard. Apply the extract and standard in small increments blowing to dryness in between to assure a tight, dense spot.

Assemble the sandwich chamber as follows: Align the vinyl spacer on the silica gel side of the plate so that it conforms with the sides and top, which have been scraped clean of silica gel. Cover with a clear glass face plate (a spare may be found with the TLC plates package) and hold the "sandwich" together with binder clips as shown in Figure A-8, one at each of the side and top edges.

Fill the trough with 40 ml of chloroform (3-4 mm depth) and press on the slotted plastic cover. Place on a level, stable surface and insert the "sandwich" assembly; by proper location of the binder clips, the assembly can be made to stand upright. A small leaning forward or backward is acceptable. When the solvent reaches the line for the solvent front, remove the "sandwich" from the trough, disassemble, and allow to dry.

Break the prescored plate as follows: Turn the adsorbent surface up. Grasp the clear end of the plate with the thumb and forefinger of each hand,

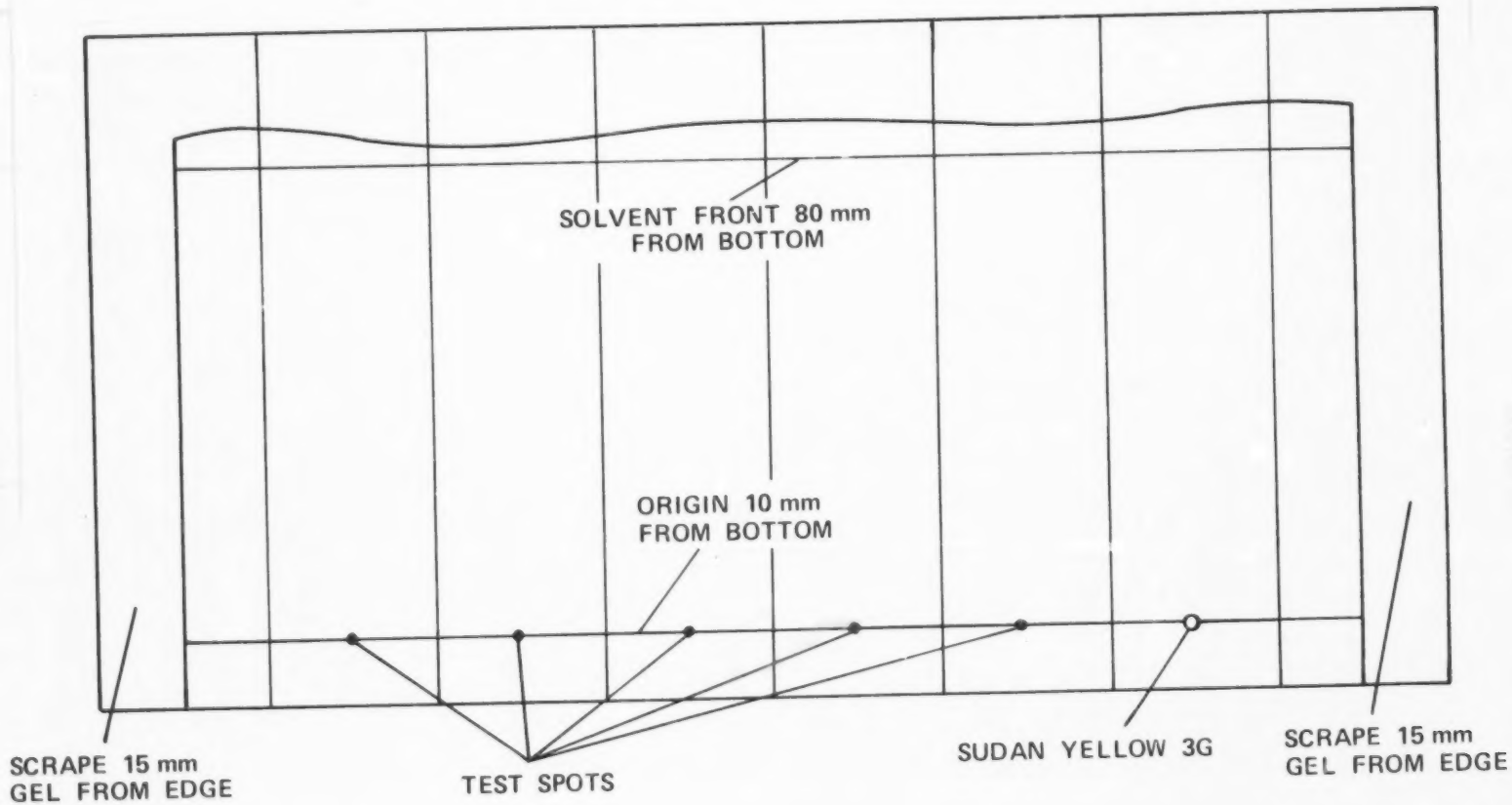

Figure A-7. Preparation of a chromatographic plate.

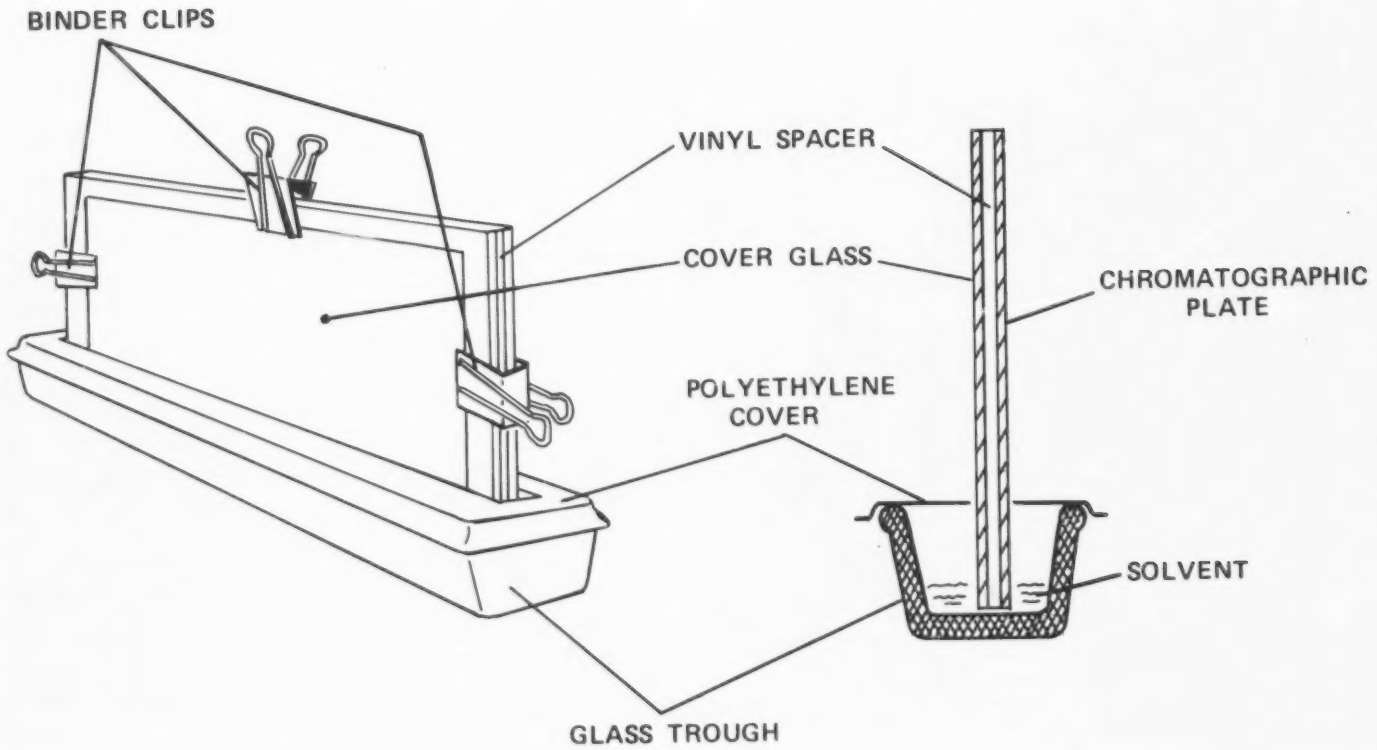

Figure A-8. Sandwich chamber assembly.

on either side of the appropriate score mark. Twist hands sharply downward to open score mark on bottom surface and snap plate.

Use the proper visualization procedures to locate any contaminants. When a contaminant is found, note its color and mark the center of its apparent mass or the leading and trailing edges of a streak. Measure these distances from the origin in mm. The R_f value is the distance moved by the material, divided by the travel of the chloroform (70 mm). The R_s value is defined as the distance traveled by the unknown, divided by the distance traveled by Sudan Yellow 3G. Record this data.

Instant Data System (IDS). The IDS system contained in the kit has cards with data for specific contaminants (Figure A-9). Data acquired at the scene of an incident may be entered on a form provided for this purpose. This information is then compared with that contained in the cards. When the number of data cards is small, this is easily done by observing the notches that are evident on the edges of the stack. However, as the card collection becomes greater, it may be desirable to use sorting needles.

Depending on the number of cards and the quality of data, it should be possible to identify some compounds specifically, and narrow others to general classes. It is evident that many factors will affect the data, e.g., age of the spill, background interference, concentration, color definition, etc. It is at these times that the skill and experience of the operator will be a significant factor.

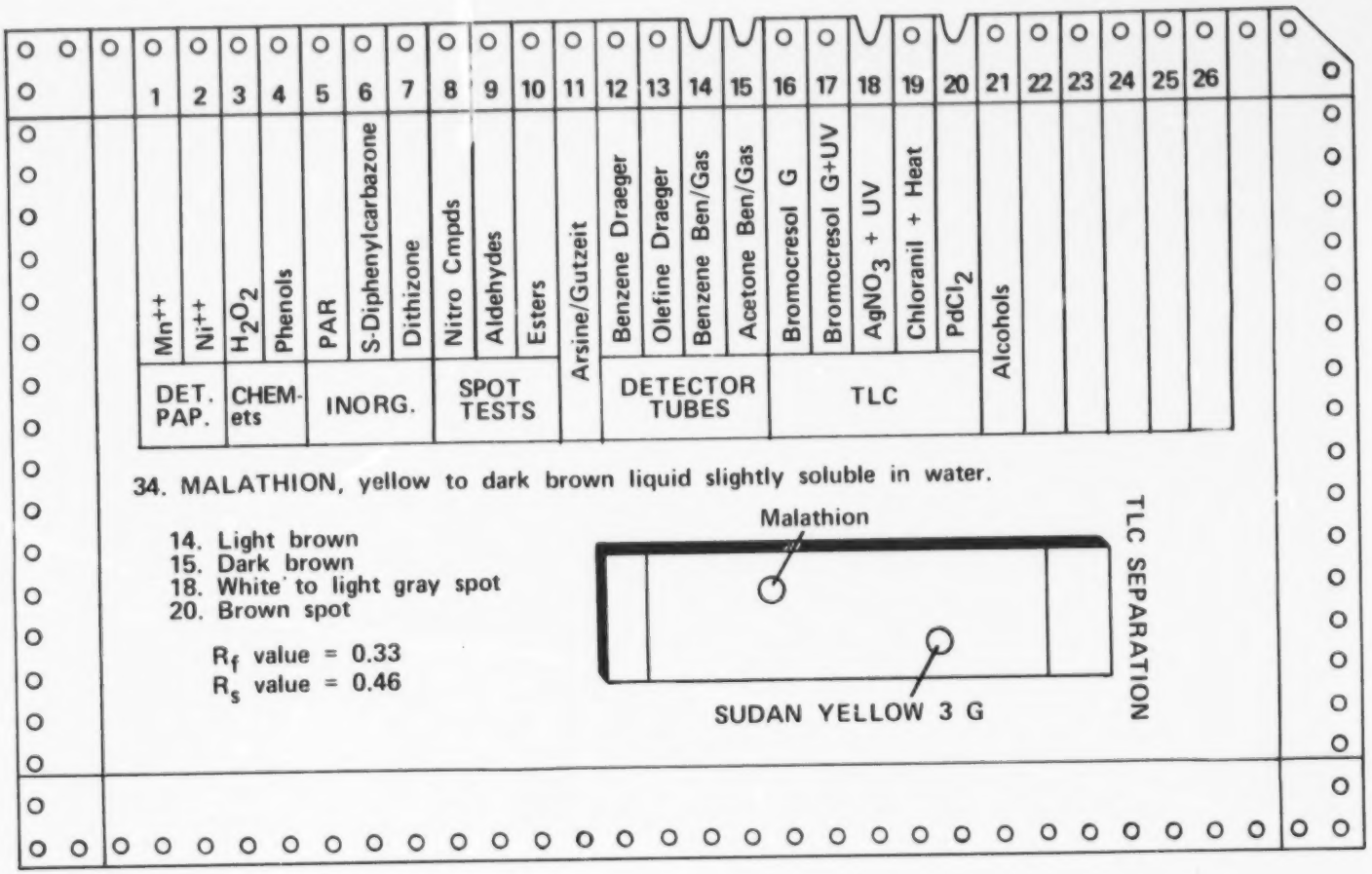

Figure A-9. IDS card prepared for identification of malathion.

SECTION 3

ANALYTICAL PROCEDURES

This section contains detailed procedures for using the ID Kit in the analysis of contaminants in water, soil, or in a neat condition. The analytical scheme is shown in Figure A-10 and the types of materials detected in Table A-1.

Before starting any analytical work, it is useful to record data regarding the contaminant, such as color, physical form, solubility if in water, odor, etc.

As the analytical data is accumulated it should be entered in the form shown in Figure A-11. When analysis is completed, the data should be compared with that contained in the data cards.

DETECTOR PAPERS

Water Samples

Test No. 1: Immerse the Mn^{++} test strip in the water sample until wet, then remove. After 15 seconds, note the color. Colors observed may range from dark blue to dark green.

Test No. 2: Immerse the Ni^{++} test strip in the water sample until wet. Remove and note the color after 1 minute. Colors may range from orange to pink to red.

Soil Samples

Prepare the sample by adding about 25 ml of loosely packed soil to the 50-ml beaker. Add distilled water to the 50-ml mark, stir well, and filter with the filter assembly. Complete tests 1 and 2 above. Hold the sample for tests by CHEMets, inorganic sprays, and spot tests.

Neat Samples

Place 0.1 gm solid (level measuring spoon) or 2 drops liquid (eyedropper) in the 50-ml beaker. Add 50 ml distilled water, stir well, and allow to stand 5 minutes. Complete tests 1 and 2 above. Hold the sample for tests by CHEMets, inorganic sprays, and spot tests.

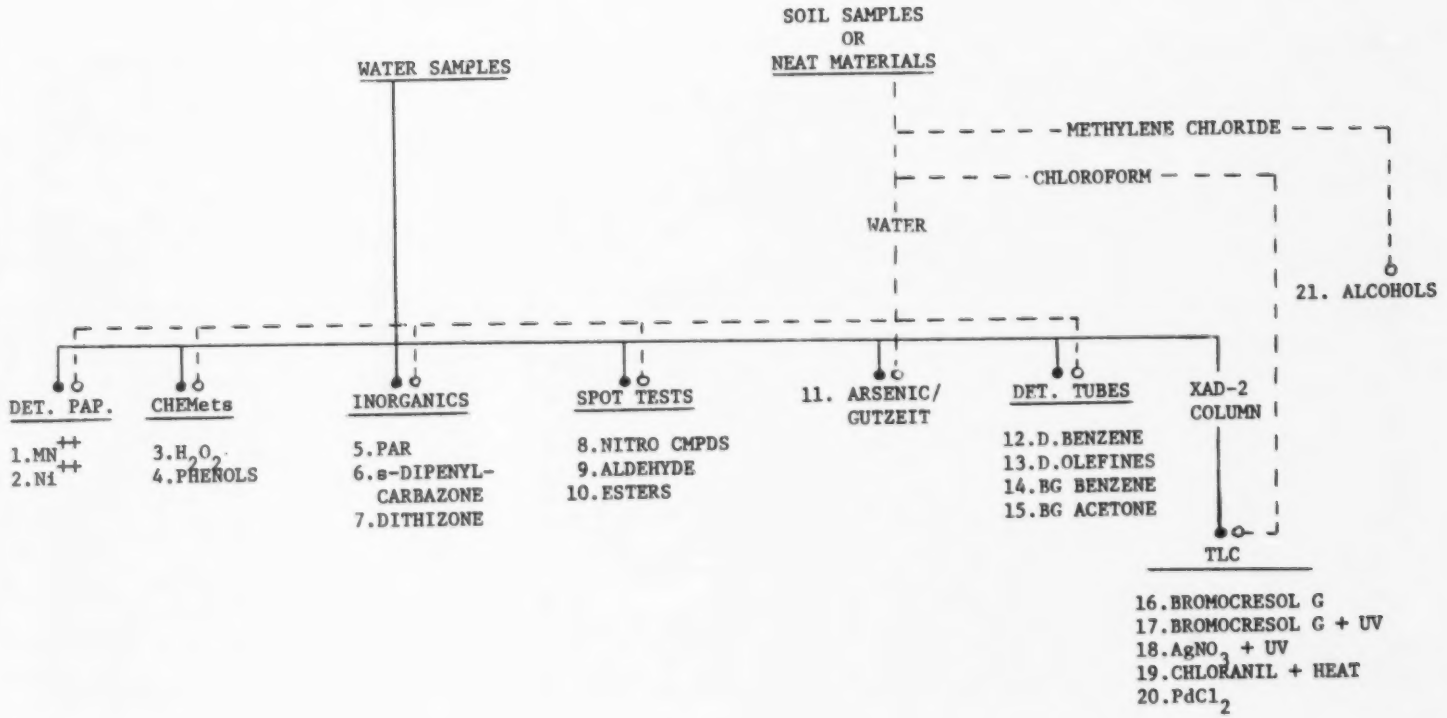

Figure A-10. Analytical scheme.

TABLE A-1. MATERIALS DETECTED BY THE ID KIT

Test Process		Test	Materials Detected
Detector Papers	1.	Mn^{++}	Certain inorganic metal compounds
	2.	Ni^{++}	Certain inorganic metal compounds
CHEMets	3.	Hydrogen peroxide	Certain inorganic metal compounds, inorganics
	4.	Phenols	Certain inorganic metal compounds, organics, phenol
Inorganics Spray Reagents	5.	PAR	Certain inorganic metal compounds
	6.	s-Diphenylcarbazone	Certain inorganic metal compounds
	7.	Dithizone	Certain inorganic metal compounds
Spot Tests	8.	Nitro compounds	Organic nitro compounds
	9.	Aldehydes	Acetaldehyde
	10.	Esters	Esters
Arsenic/ Gutzeit	11.	Arsenic/Gutzeit	Arsenic, antimony, aliphatic arsenicals
Detector Tubes	12.	Draeger benzene	Certain volatile organics
	13.	Draeger olefin	Certain volatile organics
	14.	Ben/Gas benzene	Certain volatile organics
	15.	Ben/Gas acetone	Certain volatile organics
	16.	Bromocresol green	Amines, certain organic acids
	17.	Bromocresol green + UV	Certain organochlorine materials
Thin-Layer Chroma-tography	18.	Silver nitrate + UV	Amines, organic acids, organochlorine materials, thiophosphates, phenol
	19.	Chloranil + heat	Amines, certain organic acids, phenol
	20.	Palladium chloride	Amines, acids, thiophosphates
Alcohols	21.	Alcohols	Alcohols

Test Process	Test	Response
DETECTOR PAPERS	1. Mn^{++}	
	2. Ni^{++}	
CHEMets	3. Hydrogen Peroxide	
	4. Phenols	
INORGANICS SPRAY REAGENTS	5. PAR	
	6. s-Diphenylcarbazone	
	7. Dithizone	
SPOT TESTS	8. Nitro Compounds	
	9. Aldehydes	
	10. Esters	
ARSENIC/GUTZEIT	11. Arsenic/Gutzeit	
DETECTOR TUBES	12. Draeger Benzene	
	13. Draeger Olefine	
	14. Ben/Gas Benzene	
	15. Ben/Gas Acetone	
THIN LAYER CHROMATOGRAPHY	16. Bromocresol Green	
	17. Bromocresol Green + UV	
	18. Silver Nitrate + UV	
	19. Chloranil + Heat	
	20. Palladium Chloride	
ALCOHOLS	21. Alcohols	

Figure A-11. Data sheet for ID kit.

CHEMETS

Water Samples

Test No. 3: Using a snapper, break the tip of a hydrogen peroxide ampoule beneath the surface of a water sample collected in a beaker. Invert several times to assure mixing. A response may be a brown or yellow color or even the formation of a precipitate.

Test No. 4: Place 25 ml of sample in a beaker. Stir the sample with the coated tip of a phenol CHEMet until all the crystals of potassium ferricyanide have dissolved. Place the CHEMet in the barrel of the snapper and break the tip below the surface of the water. Invert several times to assure mixing. A response may range in color from orange to brown to purple, and may even include a precipitate.

Soil Samples

Using the sample prepared for testing with detector papers, perform tests 3 and 4.

Neat Samples

Use the sample prepared for testing with detector papers and complete tests 3 and 4. Stir the sample to obtain a representative mix of suspended material.

INORGANICS SPRAY REAGENTS

Water Samples

Snap off three segments of TLC plate using the procedure described in Section 2, Equipment and Methodology, Thin-Layer Chromotography. Spot 8 microliters of water sample on each segment using a micro-pipette. The pipettes fill easily with water, but may require pressure with the rubber bulb to completely empty. It is not necessary to keep spots compact; the water may be allowed to come out in a continuous flow. Allow the samples to dry.

Test No. 5: Spray one segment of TLC plate with PAR reagent. Note the color after 5 minutes. Colors may range from orange to purple.

Test No. 6: Spray a second segment of TLC plate with s-diphenylcarbazone reagent. Note the color after 5 minutes. Colors include bright red-purple and deep purple.

Test No. 7: Spray the third segment of TLC plate with dithizone reagent. Note the color immediately. Colors can include yellow, yellow-green, orange, and red-orange.

If any results with the above tests are in doubt, they should be compared with a distilled water blank.

Soil Samples

Using the sample prepared for testing with detector papers, spot samples as described above and complete tests 5, 6, and 7.

Neat Samples

Use the sample prepared for testing with detector papers, and complete tests 5, 6, and 7. Stir the sample to obtain a representative mix.

SPOT TESTS

Water Samples

Test No. 8, Nitro compounds: Crack off a segment of TLC plate. With an eyedropper, place one drop of the water sample on the plate, followed by one drop of ethanolamine. A deep yellow color is obtained with nitro compounds. Since there is always some yellow color present due to the reagent, this test should be compared with a distilled water blank.

Test No. 9, Aldehydes: Crack off a segment of TLC plate. In turn, add the following materials: one drop of piperidine, one drop of sodium nitroprusside, and with an eyedropper, one drop of water sample. A deep purple color is obtained with aldehyde.

Test No. 10, Esters: To 1 ml of test water in a test tube, add 0.5 ml (10 drops) of hydroxylamine hydrochloride and 0.5 ml (10 drops) of sodium hydroxide. Allow to stand 2 to 3 minutes. Acidify with hydrochloric acid solution (the test solution turns purple to red to orange to yellow). Stop when the soltuion just turns yellow. Add 2 drops of ferric chloride. Esters may give a color ranging from brownish-amber to a deep violet. Compare with a distilled water blank if there is any doubt.

Soil Samples

Use the sample prepared for testing with detector papers. Complete tests 8, 9, and 10.

Neat Samples

Use the sample prepared for testing with detector papers, and complete tests 8, 9, and 10. Stir the sample to obtain a representative mix.

ARSENIC/GUTZEIT

Water Samples

Test No. 11: Add 80 ml of water sample to the effervescent jar. Prepare the stoppered cap with a Draeger Arsine Tube in place, with the arrow pointing away from the assembly. Add the contents of a Gutzeit packet to the jar and stopper quickly and firmly. Allow effervescence to continue for at least 5 minutes. A positive test is a gray purple color. Only arsenic and antimony

respond to this test. Some aromatic arsenicals may not be detected.

Soil Samples

Add 40 ml of loosely packed soil to the effervescent jar. Fill with distilled water to 80 ml and stir well. Insert the ballast between the stopper and arsine tube and follow the procedure for analysis of water (Figure A-5).

Neat Materials

Add 0.1 gm of the solid (a level measuring scoop) or 2 drops of the liquid (eyedropper) to the effervescent jar and fill to the 80-ml mark with distilled water. Stir well and continue the procedure as in the analysis of water, using the arsine tube.

DETECTOR TUBES

Water Samples

For each of the following tests, add 80 ml of water to the effervescent jar. Add one Alka-Seltzer^{R} tablet and close firmly with the cap which has the proper detector tube affixed in place. (Use the correct size Tygon tubing to match the detector tube.) Color formation on any part of the tube is considered a response. Allow the assembly to stand until effervescence is complete. Water must not be permitted to wet the contents of the detector tube. If this happens, discard the tube and repeat the test with a new one.

Tubes to be tested:

Test No. 12. Draeger Benzene Tube. Color can form on the precleanse layer or the detection portion proper. Colors can include reds, browns, blues, and purple.

Test No. 13. Draeger Olefin Tube. Color may range from light tan to brown. This tube is particularly sensitive to water and must be discarded if the crystals become wet.

Test No. 14. Bendix/Gastec Acetone Tube. Colors can include yellows, browns, greens, and blacks.

Soil Samples

Add 40 ml of loosely packed soil to the effervescent jar. Fill with distilled water to the 80-ml mark and stir well. Insert the ballast between the stopper and the detector tube and follow the procedure for analysis of water.

Neat Materials

Add 0.1 gm of the solid (a level measuring scoop) or 2 drops of the liquid (eyedropper) to the effervescent jar and fill to the 80-ml mark with distilled water. Stir well and continue the procedure as in the analysis of the water sample.

THIN-LAYER CHROMATOGRAPHY

Water Samples

Recover the contaminant from water using the procedure given in Section 2, Equipment and Methodology, Recovery from Water. Prepare a complete TLC plate as described in Thin-Layer Chromatography (Section 2) and spot with the extract from the water sample. Assemble the TLC "sandwich" chamber as described and complete the separation. Afterwards, break the plate into individual segments and spray and develop with the following reagents. Compute R_f values when spots are found, using 70 mm as the solvent travel. Also compute the R_S values by dividing the distance traveled by the unknown, by the distance traveled by the standard dye (Sudan Yellow 3G).

Test No. 16. Bromocresol green. Crack off a segment of TLC plate and spray evenly with Bromocresol green reagent. Hold the plate by the clear glass end using the plastic-tipped forceps. Spots when found will be yellow; note their colors and compute the R_f and R_S values.

Test No. 17. Bromocresol green + ultraviolet light. Take the plate prepared above and place under the UV lamp as shown in Figure A-1 of Section 2. Do not turn on the lamp yet.

Test No. 18. Silver nitrate + ultraviolet light. Assemble the silver nitrate spray reagent. Take another segment of TLC plate and spray evenly, again holding the plate with the forceps. Install the plate under the UV lamp adjacent to the Bromocresol green plate. Do not turn on the lamp yet.

Test No. 19. Chloranil. Assemble the chloranil spray reagent. Hold another segment of TLC plate with forceps and spray evenly with reagent. Place the plate on one of the heating surfaces of the inverter designated in Figure 1 of Section 2.

Hook up the inverter cable to the 12-V DC vehicular battery, paying particular attention to the polarity. Turn on the inverter and lamp switches and allow the tests to continue for 10 minutes. After 10 minutes, examine each of the three plates for spots. DO NOT LOOK AT THE PLATE OR THE LIGHT SOURCE DURING IRRADIATION, AS SEVERE EYE DAMAGE IS PROBABLE.

The Bromocresol green plate may have yellow spots in addition to those that were originally present. These must be recorded separately. Determine their R_f and R_S values.

The silver nitrate spots range from white, light browns, and grays, to dark browns and blacks. With high concentrations of some contaminants, white spots may be ringed with gray or brown. Determine the R_f/R_S values.

Test No. 20. Palladium chloride. Prepare the Palladium chloride spray assembly. Holding a segment of TLC plate with the forceps, spray it evenly with the reagent. Colors will generally be light brown. In very high concentrations, however, yellow spots with brown rings are found. Determine the R_f and R_S values.

Soil Samples

Add 10 ml of loosely packed soil to the 30-ml beaker. Add 10 ml of chloroform, stir well, and allow to settle. Filtering the extract with the filter assembly if necessary, spot the TLC plates with the extract and chromatograph and complete tests described above.

Extreme caution must be used in this test. Most organic soils will have large quantities of highly colored extractable materials. Some of these responses may be confused with those found with certain contaminants. In all cases, findings should be compared with results from other tests for possible confirmation.

When the soils are lightly colored, as in the case with some sand and clay mixes, interferences are not nearly as severe.

Neat Materials

Using a micropipette, estimate a solution of 10 microliters of liquid contaminant in 10-ml chloroform. In the case of solid samples, estimate 10 mg of unknown (spatula tip) in 10 ml of chloroform. In either case, use the solution to spot the TLC plates and complete the tests described above.

ALCOHOLS

This analysis is applicable only to soil samples and neat materials.

Soil Samples

Add 10 ml of loosely packed soil to the 30-ml beaker. Add 10 ml of methylene chloride, stir well, and allow to settle. Filter if necessary with the filter assembly.

Test No. 21. To 1 ml of the methylene chloride extract, add 2 drops of 1% vanadium (V) 8-hydroxyquinoline and 2 drops of acetic acid. After 1 minute a deep red color develops in the presence of alcohols. Again, as with the chloroform extraction described earlier, care must be taken with soils containing organic materials, as interferences may be found.

Neat Samples

Using a micropipette, estimate a solution of 10 microliters of liquid contaminant in 10 ml of methylene chloride. In case of solid samples, estimate 10 mg of unknown (spatula tip) in 10 ml of methylene chloride. Complete Test No. 21.

SECTION 4

SUPPLEMENTARY IDENTIFICATION WITH THE HAZARDOUS MATERIALS DETECTOR KIT

The ID Kit is designed to function completely independently. However, if the EPA Hazardous Materials Detector Kit is available, additional information may be gained to support identification of various materials.

If time is available, the best approach is to run through the test procedures in the detection kit. Overall, it is more sensitive than the ID Kit, and as such can serve as a guide to optimum sampling location. (It will be remembered, however, that the detection kit is designed solely for water sampling and analysis.)

When contamination is indicated by the detection kit, analysis is then completed with the ID Kit -- paying particular attention, but not limiting testing, to those procedures referenced in Table A-2. This is not to suggest that if all tests with the detection kit are negative, that contamination may be presumed to be absent. The detection kit can serve as a guide in exploiting certain tests in the ID Kit; however, all potential contaminants have not been tested with either kit. Obviously, there may be situations in which a compound undetectable by the detection kit may be found with the ID Kit.

It is suggested that if both kits are going to be used, tests for phenol and heavy metals in the detection kit be omitted in favor of tests 4, 5, 6, and 7 in the ID Kit. The tests in the ID Kit are generally simpler to perform and more reliable.

TABLE A-2. SUPPLEMENTARY IDENTIFICATION WITH THE HAZARDOUS MATERIALS DETECTOR KIT

Positive Response to Detector Kit Test for:	Remarks
Cholinesterase	Could indicate very acidic materials (confirm by pH meter). Possible organic inhibitor; check tests 12 through 20 in ID Kit.
Benzene	Generally volatile organics; check tests 3, 4, 8 through 9, and 12 through 20 in ID Kit.

(continued)

TABLE A-2 (Continued)

Positive Response to Detector Kit Test for:	Remarks
Heavy Metals	Omit. Use tests 1 through 7 and 11 in ID Kit.
Phenol	Omit. Use test 4 in ID Kit.
Cyanide	Blue color response is definite for cyanide. Other colors may indicate organic or inorganic contamination.
pH	Could indicate organic or inorganic contamination.
Conductivity	General indicator of inorganic materials.
Nitrate N_2	May indicate organic or inorganic materials.
Color	Could be organic or inorganic materials. However, in TLC procedures, organic materials will generally show some movement, whereas the inorganics will remain at the origin.
Sulfate	This test is very selective for sulfate. No interference is known.
Phosphate	May indicate phosphates, arsenates, arsenites, and bromates. Arsenic may be confirmed by test 11 in the ID Kit.
Ammonia, N_2	May indicate a wide range of organic and inorganic materials containing nitrogen.
Chloride	May indicate a wide range of organic and inorganic materials.
Fluoride	Fairly selective for fluoride compounds, unless very high concentrations of other materials are present.
Turbidity	May indicate a wide range of low solubility organic or inorganic compounds.

SECTION 5

CONSUMABLE MATERIALS

Cat. No.	Description	Unit	Unit Price ($)	Source
P-1126-4	Quant Dip Strip, EM Labs, Manganese	pkg/10	9.25	Scientific Products 8855 McGraw Road Columbia, MD 21045
P-1126-5	Quant Dip Strip, EM Labs, Nickel	pkg/10	9.25	Scientific Products
	Chemetrics, Type HP for Hydrogen Peroxide	pkg/30	15.00	Chemetrics, Inc. Mill Run Drive Warrenton, VA 22186
	Chemetrics, Type P-12 for Phenols	pkg/30	15.00	Chemetrics, Inc.
	PAR: 0.63 gm 4-(2 Pyridylazo) resorcinol in 1-liter methanol			Spray cans may be prepared by Case-Mason Filling, Inc., Joppa, MD 21085
	s-Diphenylcarbazone: 5 gm in 1 liter chloroform			Laboratory
	Dithizone: 0.5 gm Diphenylthiocarbazone in 1 liter chloroform			Laboratory
5-718-50	Gelman Chromist propellant	pkg/5	17.00	Fisher Scientific Co. 7722 Fenton Street Silver Spring, MD 20910
D-1008	Microliter pipettes, 8 microliters	vial/100	5.00	Analtech, Inc. 75 Blue Hen Drive Newark, DE 19711

Cat. No.	Description	Unit	Unit Price ($)	Source
10521	Hardlayer (organic binder) TLC Uniplates, Silical Gel HL, Pre-scored	box/25	40.00	Analtech, Inc.
	Gutzeit Bags	ea	0.50	Midwest Research Institute 425 Volker Blvd. Kansas City, MO 64110
CH 25001	Draeger detector tube, Arsine 0.05/a	box/10	17.00	National Mine Service Co. 3001 Koppers Bldg. Pittsburg, PA 15219
6718801	Draeger detector tube, Benzene 5/a	box/10	17.00	National Mine Service Co.
CH31201	Draeger detector tube, Olefins 0.05%/a	box/10	16.00	National Mine Service Co.
121	Bendix/Gastec detector tube, Benzene	box/10	8.48	Bendix Corp., Environmental & Process Instr. Division P.O. Box 831 Lewisburg, WV 24901
151	Bendix/Gastec detector tube, Acetone	box/10	8.48	Bendix Corp., Environmental & Process Instr. Division
	Alka-Seltzer^{R} (without aspirin)	box/20	0.80	Drugstore
3500010-1	Sample Reservoirs	pkg/100	16.75	Brinkmann Instruments, Inc. Cantiague Road Westbury, NY 11590
3500021-6	Absorbent Cartridges	pkg/100	45.00	Brinkmann Instruments, Inc.
3500030-5	Filter Cartridges (snip off lower tips to fit carrying case)	pkg/100	32.00	Brinkmann Instruments, Inc.

Cat. No.	Description	Unit	Unit Price ($)	Source
6801150-7	Bromocresol Green, aerosol can	ea	12.00	Brinkmann Instruments, Inc.
61693	Prefilters, Type A Glass Fibers, 25 mm	pkg/500	15.00	Gelman Instrument Company 600 So. Wagner Road Ann Arbor, MI 48106
	Silver Nitrate: 5 gm in 1 liter water			Laboratory
	Chloranil: 5 gm in 1 liter chloroform			Laboratory
	Palladium Chloride: 5 gm in 1 liter methanol			Laboratory
	Sodium Nitroprusside: 5% in water			Laboratory
	Sodium Hydroxide: 12.5% in methanol			Laboratory
	Hydroxylamine Hydrochloride: 12.5% in methanol			Laboratory
	Hydrochloric Acid Solution: 10 ml HCl and 10 mg Sodium Allzarin Sulfonate diluted to 100 ml with water			Laboratory
	Ferric Chloride Solution: 5 gm $FeCl_3$ and 5 drops HCl in 50 ml water			Laboratory
	Vanadium (V) 8-hydroxyquinoline: 1% in methylene chloride			Laboratory

Cat. No.	Description	Unit	Unit Price ($)	Source
	Sudan Yellow 3G: 0.1% in chloroform			Laboratory
	Ethanolamine			Laboratory
	Glacial Acetic Acid			Laboratory
	Chloroform			Laboratory
	Methylene Chloride			Laboratory
	Piperidine			Laboratory
	Distilled Water			Laboratory

REFERENCES

Silvestri, A., A. Goodman, L.M. McCormack, M. Razulis, A.R. Jones, Jr., and M.E.P. Davis. Development of a Kit for Detecting Hazardous Material Spills in Waterways. Environmental Protection Series. EPA-600/ 2-78-055. March 1978.